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# OCTOBER—1949

No. 161



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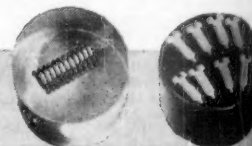
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# ASTM BULLETIN

"Promotion of Knowledge of Materials of Engineering, and Standardization of Specifications and Methods of Testing"

TELEPHONE—Rittenhouse 6-5315

R. E. Hess, Editor  
R. J. Painter, Associate Editor

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OCTOBER, 1949

## Many New Specifications and Tests Accepted

**Standards Committee Approves Actions on Ferro-Alloys, Asbestos-Cement Products, Light Metals, Glass, Gaseous Fuels, Road and Paving Materials, and Testing Methods**

**T**HE Administrative Committee on Standards held its usual fall meeting at A.S.T.M. Headquarters in Philadelphia on September 22 and accepted a large number of recommendations submitted to it by many of the Society's committees. The accompanying table lists the titles of the specifications and methods on which the Administrative Committee took action, and further on in this article will be found details as to the nature of the revisions being made and the scope of new tentatives accepted. All of the new tentatives and revisions will be published in the appropriate volumes of the 1949 Book of A.S.T.M. Standards.

Generally the Administrative Committee on Standards meets but once a year—about two months after the Annual Meeting. It can meet more often if there is need for such a meeting. (Many members may recall the special meeting of the Administrative Committee on Standards during the Annual Meeting in New York in 1945 when travel restrictions permitted the Society to hold only a General Business Session.) However, it can and does act on committee recommendations at any time during the year by letter ballot of the committee.

The Administrative Committee has for its functions broadly (1) to promote the standardization work of the Society; (2) to consider means of general policy concerning standardization activities, including relationship with similar activities of other bodies; (3) to consider the desirability of expansion of standardization work into new fields; (4) to review annually the progress in the Society's standardization work; and (5) to pass upon proposed new standards, proposed amendments of existing tentatives, or proposed amendments of existing standards offered between annual meetings of the Society in accordance with provisions of the Regulations Governing Technical Committees.

In carrying out its broad functions

### Actions by the A.S.T.M. Administrative Committee on Standards September 1949

#### New Tentatives

##### Specifications for:

- Ferroboron (A 323 - 49 T).<sup>1</sup>
- Ferrotitanium (A 324 - 49 T).<sup>1</sup>
- Asbestos-Cement Flat Sheets (C 220 - 49 T).<sup>2</sup>
- Asbestos-Cement Corrugated Sheets (C 221 - 49 T).<sup>2</sup>
- Asbestos-Cement Roofing Shingles (C 222 - 49 T).
- Asbestos-Cement Siding Shingles and Clapboards (C 223 - 49 T).
- Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 - 49 T).

##### Methods of:

- Measuring Thickness of Anodic Coatings on Aluminum by Means of the Filmeter (B 244 - 49 T).
- Sampling Glass Containers (C 224 - 49 T).<sup>2</sup>
- Test for Resistance of Glass Containers to Chemical Attack (Test B-A) (C 225 - 49 T).<sup>2</sup>
- Test for Resistance of Glass Containers to Chemical Attack (Test B-W) (C 225 - 49 T).<sup>2</sup>
- Test for Resistance of Glass Containers to Chemical Attack (Test P-W) (C 226 - 49 T).<sup>2</sup>
- Test for Water Vapor Permeability of Shipping Containers (D 1008 - 49 T).
- Determination of Specific Gravity of Gaseous Fuels (D 1070 - 49 T).
- Measurement of Gaseous Fuel Samples (D 1071 - 49 T).
- Test for Effect of Water on Cohesion of Compacted Bituminous Mixtures (D 1075 - 49 T).
- Test for Distillation Range of Lacquer Solvents and Diluents (D 1078 - 49 T).
- Photometric Determination of Cobalt in Nickel (E 39 - 49 T).
- Testing and Standardization of Etched Liquid-in-Glass Thermometers (E 77 - 49 T).
- Compression Testing of Metallic Material in Sheet Form (E 78 - 49 T).
- Photometric Analysis of Aluminum Base Alloys (E 34 - 49 T).

##### Recommended Practice for:

- Describing Form of Specimen and Direction of Testing Plastics (D 1009 - 49 T).<sup>3</sup>

<sup>1</sup> Accepted August 19, 1949.

<sup>2</sup> Accepted July 27, 1949.

<sup>3</sup> Accepted September 14, 1949.

#### Tentative Revisions of Standards

##### Specifications for:

- Structural Clay Non-Load-Bearing Tile (C 56 - 41).
- Structural Clay Floor Tile (C 57 - 39).

##### Methods of Test for:

- Compressive Strength of Hydraulic Cement Mortars (C 109 - 47).
- Determination of Bitumen (D 4 - 42).
- Hard Scoured Wool in Wool in the Grease (D 584 - 47).

##### Recommended Practices for:

- Bituminous Paving Plant Inspection (D 290 - 39).

#### Revision of Standard and Reversion to Tentative

##### Specifications for:

- Molybdenum Salts and Compounds (A 146 - 39).<sup>1</sup>

#### Revisions of Tentatives

##### Specifications for:

- Aluminum-Base Alloy Die Castings (B 85 - 48 T).
- Aluminum-Base Alloy Permanent Mold Castings (B 108 - 48 T).
- Copper-Base (Brass) Alloy Die Castings (B 176 - 48 T).
- Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 48 T).
- Structural Clay Facing Tile (C 212 - 48 T).
- A.S.T.M. Thermometers (E 1 - 48 T).

##### Methods of:

- Testing Tall Oil (D 803 - 44 T).
- Test for Chlorine in Lubricating Oils by Bomb Method (D 808 - 44 T).
- Compression Testing of Metallic Materials (E 9 - 46 T).

##### Recommended Practice for:

- Designating Significant Places in Specified Limiting Values (E 29 - 48 T)
- ##### Definition of Terms Relating to:
- Glass and Glass Products (C 162 - 48 T).
  - Adhesives (D 907 - 47 T).

#### Withdrawal of Tentative

##### Methods for:

- Sampling Molybdenum Salts and Compounds for Metallurgical Use (E 66 - 34 T).<sup>1</sup>



and its specific authority on committee recommendations the committee is interested in improving the editorial preparation and presentation of proposed methods of test and specifications, and has given considerable attention to the subject.

Committee officers presenting recommendations to the Administrative Committee indicate why the recommendations are being made; give a complete analysis of the vote in the committee; explain the negative votes which are quoted verbatim; and advise what action the committee has taken or proposes to take in order to reconcile the negative viewpoint. At its meetings the Administrative Committee on Standards offers an opportunity for those who have negative viewpoints to present their reasons in person, and for committee officers to be heard in support of committee recommendations. Negative voters on committee recommendations have a right to request such an open hearing at any time and this privilege will be accorded them.

A classification of actions which can be taken by the Administrative Committee for the Society can be summarized as follows:

(1) **Acceptance as Tentative:**

- (A) Acceptance of Tentative Revisions of Standards
- (B) Acceptance of New Tentatives
- (C) Acceptance of Revisions of Tentatives
- (D) Acceptance of Revisions of Standards that are being reverted to Tentatives (essentially acceptance of a new replacing Tentative with concurrent withdrawal of the Standard).

(2) **Withdrawals:**

- (A) Withdrawal of Tentatives
- (B) Withdrawal of Standards
- (C) Withdrawal of Tentative Revisions of Standards

*Ferro-Alloys:*

Immediately after the Annual Meeting, Committee A-9 on Ferro-Alloys presented several recommendations to the Administrative Committee on Standards which were accepted on August 19. These included two new tentative specifications: Ferroboration (A 323-49 T) and Ferrotitanium (A 324 - 49 T) as well as a complete revision and reversion to tentative of the specifications for Molybdenum Salts and Compounds which will now be designated A 146 - 49 T. These are the first recommendations to come from Committee A-9 since the war.

The first two new specifications cover ferro-alloys not previously included in A.S.T.M. specifications and provided for six grades of ferrotitanium and four grades of ferroboration. The revision of

A 146 includes provisions for molybdenum oxide in bags or cans and molybdenic oxide briquets, not previously covered in these specifications. The sampling procedure now in E 66 - 34 T is being incorporated in A 146. This method was originally written by Committee A-9 and designated A 156 - 34 T. Jurisdiction was transferred to Committee E-3 and the method was renumbered E 66. Jurisdiction was returned to Committee A-9 and it is now being incorporated in A 146 and E 66 is withdrawn.

*Die-Casting:*

Committee B-6 on Die-Cast Metals and Alloys recommended changes in two of its tentative specifications for die castings. The first related to the Tentative Specifications for Aluminum-Base Die Castings (B 85) and introduced a reference to tension test specimens to be made in accordance with Fig. 8 of Standard Methods of Tension Testing of Metallic Materials (E 8) and impact specimens in accordance with Fig. 5 of the Tentative Methods of Impact Testing of Metallic Materials (E 23). A similar change was made in the Specifications for Copper-Base (Brass) Alloy Die Castings (B 176) which had previously shown the sketches of the test specimens instead of referencing them in the other specifications.

In three other specifications under the jurisdiction of Committee B-6 where physical properties are not a part of the specification requirements but are contained in the appendix as information only, an editorial change was made to indicate that the physical properties shown were obtained by use of the type of test specimens mentioned above. The specifications involved are those for Zinc-Base Alloy Die Castings (B 86 - 48); for Magnesium-Base Alloy Die Castings (B 94 - 48 T); and for Lead and Tin-Base Alloy Die Castings (B 102 - 48).

*Aluminum Specifications:*

Committee B-7 on Light Metals and Alloys presented a Tentative Method of Measuring Thickness of Anodic Coatings on Aluminum by Means of the Filmeter. This will be designated B 244 - 49 T. Several years ago, an instrument called a filmeter was introduced commercially. It is suitable for measuring, non-destructively, nonmetallic coatings on aluminum. Subcommittee VI of Committee B-7 experimented with the instrument as a means of determining the thickness of oxide coatings on aluminum and concluded that the instrument was useful if properly used. The new tentative method is an effort to promote standardized procedures for using the filmeter.

The committee also recommended minor changes in two of its specifications. In the Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108) the copper range for Alloy CS21 was increased from 9.0 to 10.0 per cent to a range of 9.0 to 11.0 per cent. The present specification for Aluminum-Base alloys in Ingot Form (B 179 - 49 T) currently provides for a common grade of ingot for making die castings of Alloys SC2 and SC5 and similarly a common grade for SC6 and SC7. For die castings, however, a higher level of zinc is permitted in Alloys SC2 and SC7 than in Alloys SC5 and SC6. The revision of B 179 will recognize this situation by separating the zinc limits for ingot of each grade into two grades so that ingot for making die castings of Alloys SC2 and SC7 can be furnished with a zinc content of 0.9 per cent maximum.

*Cement Mortars:*

The tentative revision of the Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (C 109 - 47) submitted by Committee C-1 on Cement was written in order to clarify and improve details of procedure and apparatus. The principal changes are in the requirements for mixing bowls and in the procedures for determination of flow and in applying load to the test specimens.

*Glass:*

A number of recommendations proposed by Committee C-14 on Glass and Glass Products were accepted by letter ballot vote of the Standards Committee on July 27. These include the incorporation in the Tentative Definitions of Terms Relating to Glass and Glass Products (C 162 - 49 T) the terms included in the Glass Glossary compiled by the Committee on Classification, Nomenclature and Glossary of the American Ceramic Society. This change adds a great many new terms to C 162 and results in a revision of three of the present terms and an editorial revision in others; it will accomplish a clarification and bring about uniformity of definitions of terms used by the industry.

The committee also proposed three new methods of test for resistance of glass containers to chemical attack. Test "B-A" (C 225) is intended for an evaluation of the resistance of glass containers to chemical attack, particularly when they are to be used for products having a pH less than 5.0. Test "B-W" (C 226) is intended for an evaluation of the resistance of glass containers to chemical attack, particularly when they are to be used for products having a pH of 5.0 or more. Test "P-W" (C 227) is



intended for an evaluation of the chemical resistance of glass containers of too small capacity to allow measurement of solubility of the unbroken bottle (ampoules, for example) by Test B-W or B-A.

#### *Structural Clay Tile:*

Committee C-15 on Manufactured Masonry Units submitted to the Standards Committee a revision of its Tentative Specification for Structural Clay Facing Tile (C 212) which added requirements for tile for plaster base finish not previously covered in the specification. In this specification also the designation "heavy" as applied to tile has been changed to "special." It is the thought of the committee that the word "special" more aptly describes the unit, as the word "heavy" implies that the unit is to be used where heavy loads are encountered. Actually this type of tile is designed for superior resistance to water penetration, higher lateral strength, and resistance to impact.

Tentative revisions were also presented to apply to the Standard Specifications for Structural Clay Floor Tile (C 57 - 39) and for Structural Clay Non-Load-Bearing Tile (C 56 - 41). In both cases the tentative revisions would delete from the specifications any requirement for absorption limits. Absorption limits contained in present specifications were primarily set to control durability requirements; however, low limits were unnecessary. Absorption is a factor in fire resistance but is only one of several such factors as cell construction, shell thickness, etc. The committee feels that minimum absorption requirements are unnecessary from a structural standpoint. If fire resistance qualities are to be evaluated in the specification, separate requirements should be set up.

#### *Asbestos-Cement Products:*

Committee C-17 on Asbestos-Cement Products is one of the newer committees of the Society, having been established just three years ago. Current acceptances by the Standards Committee are the first specifications that the committee has prepared. The four new specifications cover Asbestos-Cement Flat Sheets (C 220); Corrugated Sheets (C 221); Roofing Shingles (C 222), and Siding Shingles (C 223).

All of these represent new industry standards and serve as a guide to the purchasers, users and producers, and their scope covers asbestos-cement products for use on buildings, including residential, agricultural, commercial, and institutional types of buildings. Requirements for composition and manu-

facture, physical properties, sampling, preparation of test specimens, methods of test, shapes, sizes and dimensions, workmanship and finish, nails and fasteners, underlayment sheet, packing and shipping, and inspection are included.

#### *Lacquer Solvents and Diluents:*

Committee D-1 on Paint, Varnish, Lacquer and Related Products prepared the new Tentative Method of Test for Distillation Range of Lacquer Solvents and Diluents, D 1078 - 49 T. This method describes a procedure for determining the distillation range of pure or nearly pure compounds, or mixtures having boiling ranges between 30 and 250 C. It is intended for application to aromatic aliphatic hydrocarbons, lacquer solvents such as alcohols, ketones, esters, and similar organic compounds. (This method is not intended to be used for mineral spirits and similar petroleum solvents.)

#### *Lubricating Oils:*

A revision of the Tentative Method of Test for Chlorine in Lubricating Oils by the Bomb Method (D 808 - 44 T) was prepared by Committee D-2 on Petroleum Products and Lubricants. The current text of the method contains alternate sections for the determination of chlorine by volumetric and gravimetric procedures, respectively. On the basis of a cooperative program, the volumetric procedure was eliminated in the revised text as unnecessary and inferior to the gravimetric procedure with respect to reproducibility. The revised text also includes extensive editorial changes which were made for the sake of clarity.

#### *Shipping Containers:*

The Tentative Method of Test for Water Vapor Permeability of Shipping Containers (D 1008 - 49 T) prepared by Committee D-10 on Shipping Containers covers two procedures for determining the water-vapor permeability of bulk shipping containers, as follows: Procedure A for reclosable containers, and Procedure B for containers not designed for reclosing after once opening.

The tests may be applied as packed, or after one or more performance tests such as drum, vibration, drop, or actual shipping tests, as required. For small shipping containers requiring greater accuracy in weighing, the procedure in D 895 - 47 T can be used.

#### *Gaseous Fuels:*

Standardized methods for the determination of specific gravity are most important in the testing of gaseous fuels.

This fact was fully recognized by Committee D-3 when it undertook its program for the development of methods of testing of gases used as commercial fuels. Preparation of the methods required were considered by this committee to constitute one of its principal responsibilities. The new tentative methods, which will be designated D 1070 - 49 T, will provide a most useful tool because of their complete coverage. They furnish means for measuring the specific gravity of gaseous fuels, including liquefied petroleum gases, in gaseous state at normal temperatures and pressures. The apparatus selected is sufficiently varied in nature so that one or more of the methods specified may be employed for laboratory, control, reference, or any purpose where it is desired to know the relative density of a gas or gases as compared to the density of dry air at the same temperature and pressure.

The direct weighing method is specified for reference test purposes and for calibration; it should be employed (1) for determinations requiring exceptional accuracy, and (2) for determination of the specific gravity of gases to be used for the calibration of specific gravity instruments. As this procedure is not practical for field determinations, two acceptable methods using commercially available balances, (1) the Ac-Me Balance (4-spring type) and (2) the Arceo Anubis Recording Gas Gravitometer, were selected. In accepting the above instruments for use, it is not intended to imply that there are not other equally accurate and satisfactory instruments commercially available, or that others will not be developed in the future.

This new method is based on the assumption that fuel gases tested for specific gravity follow the ideal gas laws governing the temperature and pressure relationships of ideal gases. This assumption can be made because, within the range of temperatures and pressures used in the measurement of the specific gravity of fuel gases, any such deviation is negligible.

Methods D 1071 - 49 T for measuring gaseous fuel samples provide means for measuring quantities of gaseous fuel samples, including liquefied petroleum gases, in gaseous state at normal temperatures and pressures. The equipment selected is sufficiently varied in nature so that one or more of the methods prescribed may be employed for laboratory, control, reference, or in fact any purpose where it is desired to know the quantity of gaseous fuel or fuel samples under consideration. The unit of measurement is the standard cubic foot.

Detailed items of the committee's general assignment were as follows:

1. Development of standards for temperature, pressure, and humidity, employed in the measurement of gaseous fuel samples.
2. Analysis of various methods employed in the measurement of gaseous fuel samples showing the limitation of each.
3. Development of standard methods for calibrating apparatus used in measuring gaseous fuel samples.
4. Study of variations of gaseous fuel samples from ideal gas laws under the variations of temperature and pressure encountered during measurement and corrections to be employed to compensate for same.

In addition to the general assignments indicated there were a number of special considerations which are set forth as (1) standardization of units of measurement; (2) designation of the different types of measuring apparatus to be employed; (3) study of the accuracy of various types of meters employed for measurement purposes; (4) establishment of standard methods of calibration, and (5) development of allowable tolerances and probable errors ordinarily encountered in the measurement of gaseous fuel samples.

#### *Road and Paving Materials:*

Committee D-4 on Road and Paving Materials presented to the Administrative Committee recommendations for two new tentative methods, one new tentative specification and for tentative revisions of two standards.

There has long been a demand for a laboratory procedure for determining the effect of water and moisture on the service behavior of bituminous paving mixtures. Intensive study and work on this subject has been under way in D-4 and as a result the new Method of Test for Effect of Water on Cohesion of Compacted Bituminous Mixtures (D 1075 - 49 T) is the most significant and practical method to be developed in recent years. It is desirable to standardize the procedure both for its application in practice and for its use in research investigations. This test is intended to measure the loss of cohesion resulting from the action of water on compacted bituminous mixtures containing penetration-grade asphalts. A numerical index of reduced cohesion is obtained by comparing the compressive strength of freshly molded and cured specimens with the compressive strength of duplicate specimens that have been immersed in water under prescribed conditions. Other related methods of test are in preparation.

The Tentative Method of Test for Compressive Strength of Bituminous Mixtures (D 1074 - 49 T) is one of several methods in preparation on physical test for compressed bituminous mixtures. It is an essential part of the proposed method of test for effect of water on cohesion. Standardization of procedure is necessary for application of these testing methods in research and in practical engineering use. This method of test for compacted bituminous mixture of the hot-mixed, hot-laid type for use in roadway surfaces and base courses is intended to give an index of the stability of resistance to displacement of such mixtures when properly compacted in the roadway. Since the test is made on molded cylindrical specimens unsupported laterally, the indexes or test values obtained are conservative, being materially less than the actual stability or resistance to displacement of the same mixtures in place in the road.

The new Tentative Specifications for Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 - 49 T) are intended to replace the former Standard Specifications for Sand for Sheet Asphalt and Bituminous Concrete Pavements (D 162 - 29), which were withdrawn in 1949. Terminology and requirements of the former standard specification have been changed to be consistent with tentative specifications for asphaltic paving mixtures (D 947 - 48 T and D 978 - 48 T). These new specifications cover the requirements for fine aggregate for use in sheet asphalt and bituminous concrete pavements. It is recognized that for certain purposes satisfactory results may be obtained with other materials. In such cases the use of fine aggregate not conforming to the specifications may be authorized under these specifications only under special provisions based on field experience or laboratory studies of the possibility of designing a mixture of materials to be used on the job. The mixture must yield sheet asphalt or bituminous concrete equivalent in quality to the specified mixture made with materials complying with these specifications in all respects.

In the Tentative Revision of Standard Method of Test for Determination of Bitumen (D 4 - 42), Method No. 2 of the present standard has been revised to reduce the time required for completing determinations of bitumen in materials containing fine mineral matter. The standard has also been revised editorially to clarify details of procedure and improve the phraseology. The Tentative Revision of Recommended Practice for Bituminous Paving Plant Inspection (D 290 - 39) is mainly editorial in nature, intended to bring the termi-

nology into accord with present practice and to make suitable reference to A.S.T.M. standards and tentatives approved in recent years.

#### *Textiles:*

The Tentative Revision of the Standard Method of Test for Hard Scoured Wool in Wool in the Grease (D 584 - 47), prepared by Committee D-13 on Textiles, provides greater accuracy by re-writing Section 7 on Ash to specify a 5-g. rather than a 3 to 5-g. portion; caution against loss of mineral matter; and use of a muffle furnace for ignition rather than an open crucible. The revised section reads: "An approximate 5-g. portion, accurately weighed, of oven-dry scoured wool from each test specimen and from the tags shall be taken, avoiding the loss of mineral matter. Each portion shall be placed in a tared porcelain or vitreous silica dish, approximately 85 mm. wide and 34 mm. deep, and charred over a burner until no more volatile matter is produced. The charred specimens shall be ignited in a muffle furnace at approximately 700 C. to constant weight. NOTE.—Wool is likely to swell and froth when heated. To avoid loss during charring, the dish should be heated at one side initially."

#### *Adhesives:*

The revision of the Tentative Definitions of Terms Relating to Adhesives (D 907) prepared by Committee D-14 on Adhesives will add 49 new definitions to the 22 now contained in this document. Among the new definitions being added are definitions for curing time, drying temperature, glue, laminate, mucilage, paste, pressure-sensitive adhesive, setting temperature, setting time, sizing, thermoplastic, thermoset, wet strength, tack range, and vulcanization.

#### *Naval Stores:*

Reviewing the various methods under its jurisdiction during the Annual Meeting in Atlantic City, Committee D-17 on Naval Stores decided to incorporate an electrometric titration procedure for the determination of rosin acids number in the Tentative Methods of Testing Tall Oil (D 803 - 44 T). At the same time the modified Wolff method and the McNicoll method were revised for alternate procedures. The method for determination of acid number was rewritten with referee and alternate procedures; also rewritten were the methods for saponification number. Those parts of the methods relating to unsaponifiable matter and fatty acids were revised and alternate methods were added for viscosity and color.



### Plastics:

It is essential for an industry to have common terms and classifications coded in a standardized manner so that when test values are reported they will properly describe the form of the material tested. That was the basic reason for the development by Committee D-20 on Plastics of the Tentative Recommended Practice for Describing Form of Specimen and Direction of Testing Plastics (D 1009 - 49 T) which was accepted by the Standards Committee on September 14, 1949. This recommended practice gives a simple code of letter designations to describe the form of material tested and the direction of stress application.

### Methods of Testing:

Committee E-1 prepared two new tentative methods of test. One of these covered the Testing and Standardization of Etched Stem Liquid-in-Glass Thermometers (E 77 - 49 T). This method describes the principles, apparatus, and procedures for visual and dimensional inspection, test for permanency of range, and standardization of liquid-in-glass thermometers. It is intended to apply to A.S.T.M. thermometers as well as to liquid-in-glass thermometers in general.

The second new tentative covers the Compression Testing of Metallic Materials in Sheet Form (E 78) which, while intended to cover the compression testing of metallic materials in sheet form, may also be desirable for use with metallic materials in thin sections other than in sheet form. The data obtained from a compression test may include the proportional limit, the elastic limit, the yield strength, the yield point, and the modulus of elasticity. The ultimate compressive strength cannot be determined because failure occurs by local buckling before the ultimate compressive strength of the material is reached.

The last-mentioned test method required a revision of the Tentative Methods of Compression Testing of Metallic Materials, E 9 - 46 T, to delete the material now contained in E 78. This will also involve a change in title of this method to "Compression Testing of Metallic Materials in Other Than Sheet Form." The revision also clarifies Section 3 as to requirements for bearing block faces and changes Section 7 on speed of testing so as to make rate of stressing or rate of straining the specimen the only permissible primary standard of speed.

Two new types of thermometers are being added to the Tentative Specifications for A.S.T.M. Thermometers (E 1 - 48 T), and the requirements for one type

of thermometer are being revised. The new freezing point thermometer specification was requested by Committee D-16 on Industrial Aromatic Hydrocarbons. The new petrolatum melting point thermometers were requested by Committee D-2 for use in the revision of Standard Method D 127. The revisions in the bomb calorimeter thermometers involve improvements in certain manufacturing details.

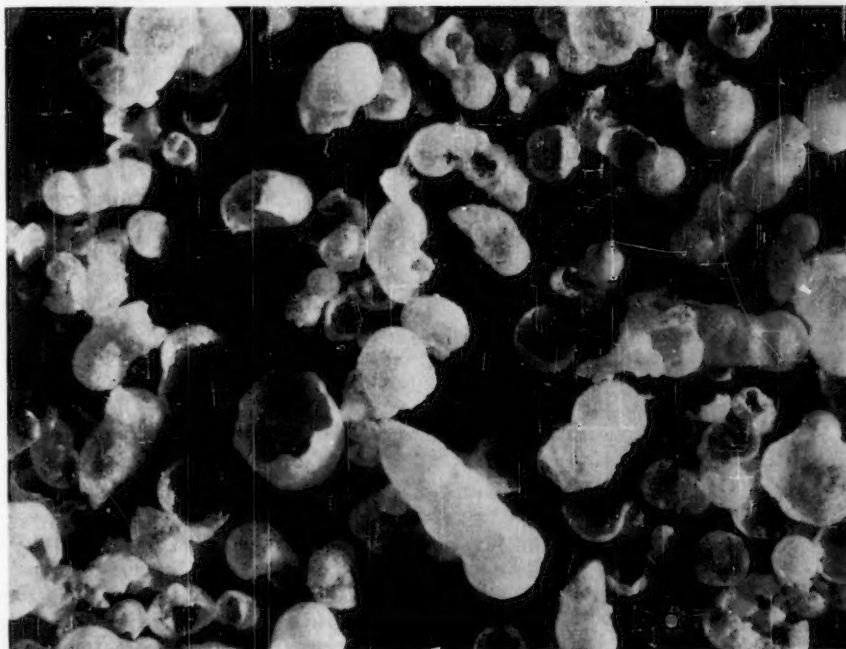
### Chemical Analysis of Metals:

Committee E-3 on Chemical Analysis of Metals prepared the Tentative Photometric Method for Determination of Cobalt in Nickel (E 39 - 49 T) which is in effect a tentative revision of the Standard Methods of Chemical Analysis of Nickel (E 39 - 45) and provides an accurate method for determining cobalt by the Nitroso-R-Salt Method which is considerably more rapid than the gravimetric procedure for cobalt that is part of Standard Methods E 39.

The committee also prepared the new Tentative Photometric Methods for the Analysis of Aluminum Base Alloys, E 34 - 49 T, which provides rapid up-to-date analytical procedures for the determination in aluminum-base alloys of manganese by the Periodate Method; chromium by the Diphenylcarbazide Method; titanium by the Hydrogen Peroxide Method; iron by the *o*-Phenanthroline Method; copper by the Hydrobromic Acid Method; and nickel by the Dimethylglyoxime Method.

### Significant Figures:

One of the points which often leads to controversy in the interpretation of specifications is how the numerical expressions of specification values should be interpreted. Committee E-11 on Quality Control has under its jurisdiction the tentative Recommended Practice for Designating Significant Places in Specified Limiting Values (E 29) which is now used by many committees as a means of specifying rules for such interpretation. This recommended practice was revised and brought up to date in 1948. But it was found after the revision that there was still one questionable point and that was how to round off values which were specified as being to the nearest 50, 5, 0.5, 0.05, etc. The new revision of the recommended practice will clarify this point. It provides, "In order to round off to the nearest 50, 5, 0.5, 0.05, etc., double the observed or calculated value, round off to the nearest 100, 10, 1.0, 0.10, etc., and divide by two. For example, in rounding off 6025 to the nearest 50: 6025 is doubled giving 12,050 which becomes 12,000 when rounded off to the nearest 100; when 12,000 is divided by two, the resulting number, 6000, is the rounded off value of 6025. In rounding off 6075 to the nearest 50: 6075 is doubled, giving 12,150 which becomes 12,200 when rounded off to the nearest 100; when 12,200 is divided by two the resulting number, 6100, is the rounded off value of 6075."



"Sprayed Dried Detergent"

Second prize-winning Photograph, Electron Micrograph and Photomicrographs Section, Photomicrographs Group, (macrographs of particles), in the Sixth A.S.T.M. Photographic Exhibit, by R. L. Gilbert, American Cyanamid Research Laboratory (100X)

## Progress on Publications

**I**N THE September issue of the ASTM BULLETIN on page 11 an extensive publication schedule for 1949-1950 was outlined. Attention is called, however, to the progress that is being made on some of the publications covered, particularly with respect to those which should be available shortly. The Year Book is already off press, as mentioned elsewhere in this BULLETIN, as well as the Bibliography on Metal Cleaning.

Of the various compilations of stand-

ards, it is expected that the compilations covering coal and coke (Committee D-5), textile materials (Committee D-13), and one on soaps and other detergents (Committee D-12) should be available before the end of October. The compilation covering standards relating to petroleum products (Committee D-2) will follow shortly. Members interested in securing copies of these special compilations will wish to consult the separate descriptions appearing elsewhere in this issue.

Of the various other special publications, the Symposium on Lubrication of Turbine Gear Equipment is now available and the Manual on Fatigue should be off press around the end of October to be followed by the Symposium on Metallography in Color, Symposium on Insulating Oils, and the Symposium on Low-Temperature Alloys. The Symposium on Bituminous Materials and the Symposium on Evaluation of Stainless Steels should be available before the end of the year.

### Symposium on Lubrication of High-Speed Turbine Gear Equipment Published

THIS symposium was presented at a meeting of Technical Committee C on Turbine Oils of Committee D-2 on Petroleum Products and Lubricants, held in Washington, D. C., in February, 1949, and has just become available in printed form. The symposium was arranged by a special committee consisting of: F. C. Linn, *Chairman*, General Electric Co.; C. D. Wilson, Allis-Chalmers Mfg. Co.; F. E. Rosenstiehl, The Texas Company; and D. F. Wilcock, General Electric Co. The book includes four papers and an introduction.

In the Introduction by F. C. Linn of General Electric Co. it is pointed out

that the proper lubrication of turbine-driven gears and worm gears which are used in turbines is of vital importance to the manufacturer, the refiner, and the customer. In the paper by R. T. Simpson of the Bureau of Ships, entitled "Lubrication of Naval Gearing" several problems are discussed such as limitations of gear dimensions for a straight mineral lubricant; increasing the load-carrying capacity of the gears by use of higher viscosity oil for gear mesh lubrication; thickness of oil film separating the teeth in a gear mesh under a given set of conditions, and others. L. J. Collins of General Electric Co. in his paper, "Developments in Gear Design and Their Lubrication Requirements," discusses purpose of a lubricant; characteristics of a gear that operates without lubrication; importance of surface finish;

and necessity of using lubricants of high viscosity, to mention a few. The paper, "Physical Concepts of the Establishment of the Lubricating Oil Wedge and Its Associated Load Carrying Capacity for the Mating Tooth Surfaces of High-Speed Gears," by Ernest K. Gatecombe, U. S. Naval Postgraduate School, places emphasis on the presentation of the physical concepts involved rather than on the mathematics of the subject matter. The last paper by A. R. Purdy of Socony-Vacuum Oil Co., Inc., entitled "Fundamentals of Worm Gear Lubrication" deals only with surface galling, which is the broad term for wear, welding, and seizure.

Members can order this 32-page symposium in heavy paper cover at the special price of 60 cents, the list price to nonmembers being 75 cents.

### 1949 Year Book Extensive

THE 1949 Year Book, just off press, and in course of distribution to the members, who have requested it, is a most extensive and interesting publication. It is the only place where one can get any kind of a true concept of the great extent of the A.S.T.M. technical committee structure, and the section on geographic distribution of the members really demonstrates that A.S.T.M. is a world-wide organization.

A large portion of the Year Book is devoted to the listing of members giving titles, connections, addresses. Compared with this section of about 150 pages is the portion devoted to committee personnel of over 280 pages.

There is pertinent information about the Society, a list of Honorary and Sustaining Members, information on fifty-and forty-year members and those deceased. There are data on officers, Charter and By-laws are included, and Regulations Governing the Board of Directors, Technical Committees, Papers, Committee Reports and Discussions, Recommendations on the Form of Standards, and data on various awards.

### New Edition of Publication on Coal and Coke Available

THE latest edition of "A.S.T.M. Standards on Coal and Coke" has recently become available. This compilation is sponsored by Committee D-5 on Coal and Coke and includes the methods of testing, specifications, and definitions of terms in this field, 28 in all. Methods of testing cover grindability, drop shatter test, tumbler test, screen analysis, sieve analysis, free swelling index, cubic foot weight, laboratory sampling and analysis, index of dustiness of coal and

coke; specifications included cover gas and coking coals, classification of coals by rank and grade, sieves for testing; and definitions of terms relating to coal and coke, gross and net calorific value, varieties of bituminous and subbituminous coals are given.

All those concerned with the production or use of coal and coke—chemists, technologists, purchasing agents, dealers, and others—should find this publication of service. It is available to members in heavy paper binding, with 164 pages, at \$1.50, the list price being \$2.

### Standards on Paper Products to be in Part 4, 1949 Book of Standards

THE following announcement will be of particular interest to those members and others who have issued instructions that Part 6 of the 1949 Book of Standards be sent to them. Originally the specifications and tests for paper and paper products, largely under the jurisdiction of Committee D-6, were scheduled for this Part, but a better grouping of the standards and a somewhat more logical arrangement has been evolved which will entail having

these specifications within the jurisdiction of Committee D-6 appear in Part 4. Part 6 of the Book will now cover electrical insulation, plastics, and rubber, and Part 4 will cover paint, wood, adhesives, shipping containers, and paper. There will be, of course, some duplication between the parts, particularly for test methods. Any members who had requested Part 6, having primarily in mind the standards on paper will wish to change this request to Part 2. !



## Latest Edition of Soaps Standards Available

THE latest compilation of "Standards on Soaps and Other Detergents" is now off press, and copies can be obtained from A.S.T.M. Headquarters. This publication includes the latest specifications and methods of test in the soap and detergents fields, some of the soaps covered being as follows: bar, chip, powdered, salt-water, milled and liquid toilet soap, solid soap, etc. Detergents include borax, soda ash, caustic soda, sodium bicarbonate, trisodium phosphate. Methods of tests cover sampling and chemical analysis of soaps, special detergents, sulfonated and sulfated oils, metal cleaning compositions, and others.

The book includes 126 pages, and is available in heavy paper cover to members at the special price of \$1.35. The list price is \$1.75.

## New Compilation of Textile Standards Issued

A NEW compilation of A.S.T.M. Standards on Textile Materials has just become available. All the specifications and test methods issued by the Society in this field are included, with many subjects covered, a few of them being as follows: woven fabrics, testing machines, fire-retardant properties, cotton fibers and yarns, sewing threads, hose and belting fabrics, tire cord, rayon and silk fabrics, fiber length, wool tops, woolen yarn, felt, asbestos cloth, glass yarn, glass fabrics, bast and leaf fibers, and many others. In addition to the standards, there are a

number of appendices included in the publication covering basic properties of textile fibers, yarn number conversion table, psychrometric table for relative humidity, proposed recommended practice for calculating number of tests to be specified in determining average quality of a textile material, proposed method of test for accelerated aging of textiles, proposed methods of test for estimating the clean wool content in wool in the grease, and others.

This latest edition includes 91 standards developed by Committee D-13 on Textile Materials, and contains approximately 586 pages. Members can obtain copies at the special price of \$3.40, the list price to nonmembers being \$4.50 per copy.

## 1949 Book of Standards

Good progress is being made in the preparation of the 1949 Book of Standards which is to be issued in six parts. It is expected that Parts 5 and probably 2 will appear first, sometime in December, the balance to appear very early in 1950. The Parts are to be made up as follows:

- Part 1—Ferrous Metals
- Part 2—Non-Ferrous Metals
- Part 3—Cementitious, Soils, Road and Waterproofing Materials
- Part 4—Paint, Wood, Adhesives, Shipping Containers, Paper
- Part 5—Fuels, Petroleum, Aromatic Hydrocarbons, Soap, Water, Textiles
- Part 6—Electrical Insulation, Plastics, Rubber

The prices of the Parts are:

- Part 1...\$10      Part 4...\$ 8
- Part 2... 8      Part 5... 10
- Part 3... 8      Part 6... 10
- To A.S.T.M. members: Parts 1, 5, and 6, \$7.50; Parts 2, 3, and 4, \$6 each.
- A Complete Set of All Six Parts, \$54; to A.S.T.M. members, \$40.50.

The Books will be mailed in accordance with the instructions on file at Headquarters, and soon an order-form will go to all purchasers of the Book, together with a prospectus so that they can place their orders on file.

The Books will be available in blue cloth binding, with a red backstrap stamped in gold. They will contain two Tables of Contents, and each Part will have a detailed Index.

## Schedule of A. S. T. M. Meetings

DATE	GROUP	PLACE
October 24	New England District	Boston, Mass.
October 27	Detroit District	Detroit, Mich.
October 28	New York District	New York, N. Y.
October 31–November 1	Committee D-20 on Plastics	New York, N. Y.
November 3	St. Louis District	St. Louis, Mo.
November 7	Western New York-Ontario District	Buffalo, N. Y.
November 17	Committee C-13 on Concrete Pipe	St. Louis, Mo.
November 28–30	Committee D-9 on Electrical Insulating Materials	Washington, D. C.
November 29	Pittsburgh District	Pittsburgh, Pa.
December 14	Philadelphia District	Philadelphia, Pa.
January 30–February 1	Committee A-1 on Steel	Philadelphia, Pa.
January 31	Philadelphia District	Philadelphia, Pa.
February 27–March 3	COMMITTEE WEEK AND SPRING MEETING	Pittsburgh, Pa.
June 26–30	53RD ANNUAL MEETING AND 9TH EXHIBIT OF TESTING APPARATUS AND EQUIPMENT	Atlantic City, N. J.

## Calendar of Society Meetings

- NATIONAL PAINT, VARNISH AND LACQUER ASSOCIATION—61st Annual Convention, October 31–November 2, Chalfonte-Haddon Hall, Atlantic City, N. J.
- AMERICAN INSTITUTE OF STEEL CONSTRUCTION—Annual Convention, October 31–November 3, White Sulphur Springs, W. Va.
- PACIFIC CHEMICAL EXPOSITION—November 1–5, Civic Auditorium, Chicago, Ill.
- FEDERATION OF PAINT & VARNISH PRODUCTIONS CLUBS—27th Annual Meeting, November 3–5, Atlantic City, N. J.
- AMERICAN SOCIETY OF MECHANICAL ENGINEERS—1949 Annual Meeting, November 27–December 2, New York, N. Y.
- AMERICAN INSTITUTE OF CHEMICAL ENGINEERS—Annual Meeting, December 4–7, Hotel William Penn, Pittsburgh, Pa.
- AMERICAN ASSOCIATION FOR ADVANCEMENT OF SCIENCE—Annual Meeting of Chemistry Section, December 26–31, New York, N. Y.
- AMERICAN STATISTICAL ASSOCIATION—American Society for Testing Materials—Joint Meeting, December 28, New York, N. Y.
- AMERICAN STATISTICAL ASSOCIATION—The American Society of Mechanical Engineers—Joint Meeting, December 28, New York, N. Y.
- AMERICAN CHEMICAL SOCIETY—Division Industrial and Engineering Chemistry Symposium on Absorption and Extraction, December 29–30, Columbus, Ohio.
- AMERICAN SOCIETY OF CIVIL ENGINEERS—Annual Meeting, January 17–19, 1950, New York, N. Y.
- AMERICAN INSTITUTE OF MINING AND METALLURGICAL ENGINEERS—Annual Meeting, February 12–16, Pennsylvania (Statler) Hotel, New York, N. Y.
- American Society for Testing Materials—Committee Week and Spring Meeting, February 27–March 3, 1950, Hotel William Penn, Pittsburgh, Pa.
- American Society for Testing Materials—53rd Annual Meeting and 9th Exhibit of Testing Apparatus and Related Equipment, June 26–30, Hotel Chalfonte-Haddon Hall, Atlantic City, N. J.



# ASTM Bulletin

OCTOBER 1949

NO. 161

NINETEEN-SIXTEEN  
RACE STREET  
PHILADELPHIA 3, PENNA.

U. S. Bureau of Reclamation,  
Denver

## PETROLEUM LUNCHEON

*Toastmaster:* H. G. Vesper, President,  
California Research Corp., San Francisco

*Guest Speaker:* E. A. Evans, President,  
The Institute of Petroleum (Great Britain), London

## PAINT LUNCHEON

*Toastmaster:* G. J. Grieve, Technical  
Director, Pacific Paint and Varnish  
Co., Berkeley

*Guest Speaker:* G. B. Gillin, Superin-  
tendent, San Francisco Mint on "Mak-  
ing Money the Hard Way"

## WOOD LUNCHEON

*Toastmaster:* T. K. May, Director of  
Technical Service, West Coast Lum-  
bermen's Assn., Portland

*Guest Speaker:* J. J. Gould, Consulting  
Engineer, San Francisco on "Timber  
Research Problems"

AS THIS BULLETIN goes to press, the Pacific Area Meeting is in progress. Details of the program were mailed to all members with a 16-page announcement; a rather complete write-up will appear in the December issue. In addition to the fifteen formal technical sessions, there were several sessions of papers developed by such technical committees as Petroleum Products and Lubricants, Concrete and Concrete Aggregates, Structural Sandwich Constructions, and Wood. In addition to these sessions, there were a number of meetings of technical committees and an interesting program of entertainment.

One of the features of the program that will be of interest to almost everyone at the meeting will be the Industry Luncheons that have been planned. A list of these luncheons with the presiding officers and guest speakers is shown below. This is the first time that luncheons of this kind have been features of A.S.T.M. meetings, and the outstanding men who will participate assure that each luncheon will be a most interesting affair.

## SOILS LUNCHEON

*Toastmaster:* R. F. Blanks, Chief, En-  
gineering Control and Research Divi-  
sion, U. S. Bureau of Reclamation,  
Denver

*Guest Speaker:* Evan A. Mattison, Ex-  
ecutive Vice-President, Bank of Amer-  
ica, San Francisco, on "Growth of  
the West and Branch Banking"

## CEMENT LUNCHEON

*Toastmaster:* J. E. Jellick, Manager,  
Portland Cement Information Bu-  
reau, San Francisco

*Guest Speakers:* A. Allan Bates, Vice-  
President, Portland Cement Assn.,  
Chicago. R. F. Blanks, Chief, En-  
gineering Control and Research Divi-

## '50 Nominating Committee

IN ACCORDANCE with the By-laws, providing that the Board of Directors shall select a nominating committee for officers, the Board has considered the report of the tellers, Gustaf Soderberg, of Graham, Crowley and Associates, 473 York Road, Jenkintown, Pa., and Percival Theel, Philadelphia Textile Institute, School House Lane and Henry Ave., Philadelphia 44, Pa., on the recommendation of members for appointees on the nominating committee and alternates:

### NOMINATING COMMITTEE

#### Members

E. F. Lundeen, Inland Steel Co.  
L. B. Jones, Consulting Engineer  
Giles E. Hopkins, Wool Bureau  
Frank H. Jackson, Bureau of Public Roads  
Robert Burns, Bell Telephone Labora-  
tories, Inc.  
George F. Harnden, General Electric Co.

#### Respective Alternates

Robert W. Steigerwalt, Carnegie-Illinois  
Steel Corp.  
Harry G. Miller, Chicago, Milwaukee, St.  
Paul & Pacific Railroad Co.  
Herbert J. Ball, Lowell Textile Institute  
Kenneth B. Woods, Purdue University  
Gordon M. Kline, National Bureau of  
Standards  
Lewis S. Reid, Metropolitan Life In-  
surance Co.

Serving on the 1950 Nominating Committee as *ex-officio* members are the three immediate past-presidents, Arthur W. Carpenter, T. A. Boyd, and Richard L. Templin.

The committee will meet sometime in March and nominate for each office—President, Vice-President, and five Members of the Board of Directors. The selections by the nominating committee will be announced to the members in the ASTM BULLETIN prior to transmission of official ballots.

## Offers of Papers for 1950

THE Administrative Committee on Papers and Publications will meet early in February to consider the papers to be published by the Society in 1950 and to develop the program of the 1950 Annual Meeting to be held in Atlantic City, N. J., June 26-30. All those who have in mind offering papers for presentation at the Annual Meeting and publication by the Society should send these offers to Society Headquarters no later than January 16.

All offers should be accompanied by a summary which should make clear the intended scope of the paper and indicate features that, in the opinion of the author, will justify its inclusion in the Annual Meeting program and publication by the Society. Suitable blanks to be used in transmitting the desired information will be sent promptly on request.

## All Items on Ballot on Standards Approved

AFTER canvassing the results for the 1949 letter ballot, the membership of the Society approved all of the items listed on the ballot. The list included 4 adoptions of new standards, 122 revisions of existing standards, 90 adoptions of tentatives as standards, and 1 amendment to the By-laws.

Details of the changes were given in the *Summary of Proceedings* which was mailed to all members in August. Of course all standards will appear in the 1949 Book of A.S.T.M. Standards which is now being prepared.



**EDITOR'S NOTE.**—This case history is second in a series to illustrate how the development of standard test methods implement research on new products and new processes. As in the first case history (see page 15 of the September BULLETIN) this information originally was submitted in a communication to the A.S.T.M. Committee on Developmental Activities. Mr. Briggs, the author, is Chairman of Section A of Cathodes, Subcommittee VIII, Metallic Materials for Radio Tubes and Incandescent Lamps, of A.S.T.M. Technical Committee B-4 on Electrical Heating, Resistance, and Related Alloys and is Electronics Engineer of The Superior Tube Co., and is in charge of that company's research and developmental work in this field. Readers of the ASTM BULLETIN will recall stories of the progress of this important work which have been published from time to time, and the article below gives an account of progress up to date.

## Test Methods and Research

By Thomas H. Briggs

THE emission of electrons from hot cathodes in vacua is the foundation of the electronics industry. Free electrons have been harnessed for practical use over a period of 40 years. Despite this fact, the fundamental theories governing their release still are not clear.

There are several excellent reasons for this: First, we are dealing with the elemental particles of mass. These can be examined only by indirect means. Second, many materials and processes must be united in perfect balance to produce a satisfactory electron tube.

It is small wonder that reliable test methods have been difficult to develop. They must be indirect, and exact control must be exercised over all save the one variable under test.

While attempting to develop yardsticks for tests which will yield reliable numerical data, research laboratories, engineering departments, and manufacturing organizations forge ahead under full power making new applications of the arbitrarily harnessed electrons and creating new demands upon the electron sources—the little-known thermionic cathodes.

In the A.S.T.M., for the past five years Section A on Cathodes of Subcommittee VIII on Metallic Material for Radio Tubes and Incandescent Lamps, of B-4 on Electrical Heating, Resistance, and Related Alloys has been studying ways in which numerical measurement can be applied to the emission characteristic as influenced by the cathode base metal. Note that this study is of the base metal alone, assuming that the important alkaline earth coating and all

of the complex ancillary processes are performed under controlled conditions. Research progresses so rapidly that test methods must always be in the process of modification, and, hence, are still tentative.

One of our landmarks of progress came when it was pointed out that actually there were many characteristics of cathodes which should be assessed separately, rather than collectively. By this concept alone it was possible to simplify the testing methods and to gain a better understanding of theory underlying cathode behavior. This point was reached only after the several subsections of the Cathode Committee had gathered masses of data for several years.

Let us cite a few examples to illustrate the present and future influence of the test methods being developed by the Cathode Committee. Because of its youth and the magnitude of the task, it is not yet possible to point to many past achievements.

1. In the mid-1930's cathode sleeves were soft and difficult to handle. A cathode strength tester was devised which was incorporated into A.S.T.M. Specification B 128-38. This instrument made possible numerical comparisons between different cathode metal processing methods. In consequence, all cathodes soon became sufficiently strong as a result of the research made possible by the tester, so that deformation during assembly was practically eliminated.

During and since the war, demands arose for cathodes which would be strong at operating temperatures (850 C.) when subjected to many types of loading and shock. It is now planned to utilize this A.S.T.M. test device, with modifications, to obtain numerical data upon hot cathodes. From comparative tests, it is expected that the cathodes can be still further improved so that they will become comparable in strength to the other parts forming the ruggedized electron tube assemblies.

2. Cathode metals are alloys comprising at least half a dozen important minor elements in a nickel base. These elements total not more than 0.5 per cent, and variations of content in even the third decimal place may be highly significant. Individual cathodes weigh as little as 20 mg. Obviously, chemical analytical methods of extreme accuracy and sensitivity with small sample size are essential. Several other A.S.T.M. committees are cooperating with Committee B-4 in projects directed toward

developing Society specifications for small sample, high accuracy, chemical analytical methods.

As this Standards work progresses, it is being employed in solving one of the industry's most pressing problems—determination of a figure of merit for cathode alloys and their successive melts, based upon chemical composition.

3. But elemental composition alone is not the answer. Even the form in which these alloying agents are present must be known with some precision. Two test methods are now under preparation by the Cathode Committee: one, for determination of oxide content of certain of the elements—relatively simple and probably valuable for product control as well as for research; the other, for determination of total gas content—by vacuum fusion of the test sample. This is complex but seemingly of value to research programs.

The data from cases 2 and 3, when combined, will, it is earnestly hoped, provide knowledge which will enable cathode metals to be evaluated numerically, for correlation with electron tube performance. From this information, it will be possible to reduce the multiplicity of existing empirical alloys, and to construct intelligently new and improved alloys.

4. Cathode base metal performance for the emission characteristic alone can only be accomplished by trial in actual electron tubes. A standardized tube is thus indicated. During the five years since the Cathode committee was organized, it has developed a diode tube which has brought out a host of interesting and valuable data. Shortly the tentative diode specification will be completed. This diode tube has not proved suitable for production control due to its cost, nor for research due to its lack of ultimate sensitivity. Nevertheless, it has been valuable to research, since it has shown which processes require the greatest degree of control, and how to achieve it.

By slight modification of the diode structure, processing and test methods, several different cathode performance characteristics are already being evaluated on a research basis. One of them we feel, can offer a new tool to chemistry, and metallurgy in determining the form in which certain minor constituent elements are present in the parent alloy. This is only in early qualitative stages. With refinements, quantitative data can be expected. This will be valuable in the field of research upon the mechanism of thermionic emission.

## Joint American Statistical Assn.-A.S.T.M. Meeting

ON WEDNESDAY, Dec. 28, 1949, at 12:30 p.m., a joint luncheon meeting of the American Statistical Association and the American Society for Testing Materials will be held in New York, N. Y., probably at the Biltmore Hotel.

The subject of the meeting, at which C. L. Warwick, Executive Secretary of A.S.T.M., will preside as Chairman, will be "Statistical Design of Engineering Tests." Grant Wernimont of Eastman Kodak and W. J. Youden of the National Bureau of Standards—both are active workers on Committee E-11 on Quality Control of Materials—will be the principal speakers. Following their talks will be a panel discussion led by two others as yet unannounced. The subject is considered a very timely one.

The A.S.T.M. has been a leader in the publicizing and introduction of statistical methods. The statistical approach is assuming ever increasing importance—especially to those concerned with quality control of materials. Because of this and also feel-

ing the need for some function which would combine a social as well as technical interest to bring the two societies with mutual interests closer together, the officials of both societies led by Merrill Flood of the A.S.A. started the necessary actions. Now as a result of much work done by a Joint Committee on Arrangements (Paul S. Olmstead, of Bell Telephone Laboratories, O. P. Beckwith of Alexander Smith and Sons Carpet Co., and A. C. Holman of Western Electric Co., Inc.) this joint luncheon meeting soon is to be a reality.

Tickets will be available early in December and can be obtained from A.S.T.M. Headquarters. A.S.T.M. members will be admitted without registry fee by showing evidence of Society membership.

At 10:00 a.m., earlier the same day, there will be another joint meeting of the A.S.A. and A.S.M.E. in the same hotel; for those of A.S.T.M. who might like to attend, the program is as follows:

As previously announced, the Annual Meeting will be in Atlantic City, June 26-30, 1950, and at that time the Ninth Exhibit of Testing Apparatus and Related Equipment will be under way. The Spring Meeting is to be held in Pittsburgh during the week of February 27-March 3.

GENERAL TOPIC: Statistical Method in Production Engineering

MODERATOR: William A. MacCrehan, New York University

### SPEAKERS:

1. Harold Engstrom, Sylvania Electric Products. Topic: Determinations of Universal Production Standards
2. Sebastian B. Littauer and Adam Abruzzi, Columbia University. Topic: The Statistical Approach to Standards of Productivity

### PANEL DISCUSSION: —

David Porter, New York University  
Walter A. Schewhart, Bell Telephone Laboratories  
William Gomberg, International Ladies Garment Workers' Union  
Ralph Presgrave, J. D. Woods and Gordon, Ltd.

## Interesting District Meetings Planned for Fall and Winter

Subjects: Quality Control; Research; Solar Heating; Man-Made Isotopes; Nuclear Power; Sources of Energy; Modern Glass. All Members and Friends Invited.

**W**ITH the coming of the fall, the various A.S.T.M. Districts, functioning in industrial centers throughout the country, are renewing their activities after a quiet summer. A number of the meetings being planned are sponsored jointly with local chapters and sections of other national engineering and professional societies. Frequently our District Councils are in a position to suggest and develop interesting programs, and because some of the topics should be of distinct interest to certain organizations, the latter frequently receive special invitations to attend the meeting, and in many cases are joint sponsors of the affairs.

Brief notes appear below on some of the interesting meetings being planned, although before this goes to press one or more of them may have been held. All members of the Society, committee members, and others interested are cordially invited to attend these sessions. At some of the meetings it is planned to have President Morrow and other prominent A.S.T.M. men meet with the districts, and in all cases the topics covered should be of distinctive interest to many A.S.T.M. members.

### Quality Control—Philadelphia—October 12:

The first of four meetings sponsored by the Philadelphia District is a joint meeting with the Philadelphia Section of the American Society for Quality Control. This will be held at the Franklin Institute on the Parkway, at 8 o'clock on October 12. Robert Burns, a member of the technical staff at Bell Telephone Laboratories, Inc., Murray Hill, N. J., will discuss "Quality Control in Materials Procurement." Mr. Burns is responsible for establishing standards of quality for organic insulating materials used by the Bell System and has spent most of his working life in preparation of methods of test and specifications aimed at the highest possible quality consistent with realistic engineering and sound economics.

The program for this meeting has been arranged by H. W. Stuart and C. R. Hutchcroft of the Philadelphia District Council in conjunction with Robert S. Inglis, A.S.Q.C. Chairman, and E. B. Haden, A.S.Q.C. Vice-Chairman. The Technical Chairman of the meeting will be Charles R. Scott, Jr.,

immediate past-chairman of A.S.Q.C. Philadelphia Section.

Last year the Philadelphia District joined with the American Society for Quality Control to sponsor a two-session meeting on Quality Control in Industry which proved to be one of the most interesting and well-attended meetings in the history of A.S.T.M. districts. This year's meeting, although a one-session, evening meeting, undoubtedly should be interesting and informative.

### Research—Philadelphia—Dec. 14:

The Philadelphia District is joining with The Franklin Institute in planning an extensive program "Research for Management." This meeting will be a two-session symposium starting at 3 p.m., in Franklin Hall on Wednesday December 14 at the Franklin Institute. The program has not been completed yet, but we are assured of having an impressive group of speakers, among which are the following: Dr. Weidlein, Director, Mellon Institute, and Professor Schilling of Pennsylvania State College will speak in the afternoon, and at the evening session Dr. Clamer, President, Ajax Metal Company, and



Dr. Swann, Director, Bartol Foundation will speak. It is planned to supplement this list by three more speakers.

A dinner will take place between the sessions with a guest speaker, and there will also be a tour of the Institute's laboratories.

The Franklin Institute and the Philadelphia Council hope to present at this meeting a picture of the need for research by business organizations and give some ideas on how much to allow for research on a budget. They also hope to bring out how to plan a research program and what research facilities are available.

Everyone is cordially invited to attend this meeting, and members in the Philadelphia District are urged to pay particular attention to the direct mail notice which will be distributed prior to this meeting.

#### *Quality and Utility in Modern Glass—Pittsburgh—November 29:*

The Pittsburgh District Council is planning to hold a meeting on November 29 at the Mellon Institute Auditorium and will have Alexander Silverman Head of the Department of Chemistry, University of Pittsburgh as their speaker. Mr. Silverman, who will cover the subject of "Quality and Utility in Modern Glass," is a well-known and very experienced speaker on the general subject of glass, having lectured on the subject before scientific societies both in the United States and Europe. This particular talk will be extensively illustrated with colored slides and various unique exhibits. The ladies, who are cordially invited to attend this meeting, should be especially interested in his collection of synthetic gems.

#### *Solar Heating—New England—October 24:*

The New England District has planned its Fall Meeting to be held on October 24 at the Northeastern University Commons in Boston. It is to be a dinner meeting with two outstanding speakers. Prof. L. B. Anderson, in charge of the Department of Architecture at the Massachusetts Institute of Technology, and Prof. A. G. H. Dietz, also of the Massachusetts Institute of Technology, in the Department of Building Engineering, will speak on two phases of this pertinent subject. Prof. Anderson will present the architectural aspects, and Prof. Dietz the engineering considerations.

Prof. E. A. Gramstorff, Program Committee Chairman for the New England District, has handled the arrangements for this interesting meeting, and

dinner reservations should be made promptly with him—dinner price is \$1.70. This meeting will get under way with dinner at 6 p.m., immediately followed by the technical session.

Members in the New England District will receive direct mail notices giving further details of this meeting.

#### *Applying Man-Made Isotopes—Detroit—October 27:*

The Detroit District has arranged a very extensive two-session symposium on Applying Man-Made Isotopes in Industry, to start at 3 p.m. in the Rackham Memorial Building on Thursday, October 27. An intervening dinner will be held with C. F. Kettering as the dinner speaker. President J. G. Morrow will also be present to speak briefly. Four technical papers are scheduled as follows:

##### *AFTERNOON—3 p.m.*

*Significant and Historical Backgrounds on Isotopes—Dr. Theodore M. Switz*  
*Tracer Elements in the Inorganic Field—Dr. Harrison Brown*

##### *DINNER—6:30 p.m.*

Charles F. Kettering; J. G. Morrow

##### *EVENING—8 p.m.*

*Tracer Elements in the Organic Field—Dr. Weldon G. Brown*  
*Cautions and Precautions in the Use of Radioactive Isotopes—Dr. Wendell C. Peacock*

Between the afternoon and evening sessions there will be demonstrations of the use of equipment in radioactive isotope work.

Dinner reservations should be made promptly with A. J. Herzig, Climax Molybdenum Co. of Michigan, Inc., 14410 Woodrow Wilson Ave., Detroit 3, Mich. Dinner price—\$3. A cordial invitation is extended to all members to attend this Detroit symposium, but be sure to make your dinner reservations early.

#### *Future of Nuclear Power—Isotopes in Industry—New York—October 28:*

One day later than Detroit, the New York District and the Metropolitan Section of The American Society of Mechanical Engineers join in presenting a similar program on Friday, October 28, at 7:30 in Room 502 of the Engineering Society's Building. Albert L. Baker, Vice-President of the Kellogg Corp., New York, will speak on the "Future of Nuclear Power." "Isotopes in Industry" will be presented by John R. Dunning of the Department of Physics, Columbia University.

Arrangements for this meeting have been made by E. P. Pitman, an active member of the New York District Council, in cooperation with V. Weaver Smith of the A.S.M.E. who will be the Technical Chairman for the evening.

#### *Major Sources of Energy—St. Louis—November 3:*

Eugene Ayres, Gulf Research and Development Company, will be the speaker at a joint meeting of the A.S.T.M. St. Louis District and the Engineers' Club of St. Louis on Thursday, November 3, at 8:15 p.m. to be held in the Engineer's Club Building. Mr. Ayres will present a condensation of his very comprehensive paper on the "Major Sources of Energy" which he presented at the annual meeting of the American Petroleum Institute.

The nine "continuous" sources of energy which he will analyze will be as follows: solar energy, waterfalls, the earth's heat, vegetation, tropic waters, tides, atmospheric electricity, wind, and the heat pump.

A dinner will be held preceding the meeting at the Camille Tea Room, 4378 Lindell Blvd., at 6:15, price \$2.50, and dinner reservations should be made promptly with John M. Wendling, Secretary of the St. Louis District Council, City of St. Louis, Municipal Testing Lab., 55 Municipal Courts Building, St. Louis 3, Mo.

A notice of this meeting, with a dinner return form, is being prepared and will be distributed shortly to all members in the St. Louis area.

#### *Informal Smoker—Western New York—Ontario—November 7:*

President Morrow's "home" district will pay its respects to him through an Informal Smoker. This is to be a strictly social affair patterned after the Smoker held by the Pittsburgh District last May. President Morrow and Executive-Secretary Warwick will attend, but there is no technical talk planned for the occasion. There will be plenty of refreshments, however, and also music and entertainment. This is strictly an A.S.T.M. affair and is planned for district members only. There will be no charge.

The Smoker will be held on Monday evening, November 7, at 8 p.m., at the Park Lane Restaurant, Delaware Avenue at Gates Circle, Buffalo.

This social affair has been spark-plugged by W. H. Lutz, past-Chairman of the Administrative Committee on District Activities, and a member of the Western New York-Ontario District. Mr. Lutz along with Dr. O. W. Ellis, District Chairman, Mr. Joseph Gentile, District Secretary, and Mr. T. L. Mayer will be the Greeting Committee.

Members of the Western New York-Ontario District will be receiving a notice of this meeting and a post card which should be signed and returned.

## TECHNICAL COMMITTEE NOTES

### Refractories and New Ceramic Whiteware Committees Hold Fall Meetings

FALL meetings were held recently by C-8 on Refractories and C-21 on Ceramic Whitewares at Bedford Springs Hotel, Bedford, Pa. These meetings were both held on September 22 in conjunction with the fall meetings of the Refractories, Whitewares and Material and Equipment Divisions of the American Ceramic Society.

**Committee C-8** held an advisory meeting in the morning followed by a main committee meeting in the afternoon. Some of the subcommittees had held informal meetings previously. At the well-attended main committee meeting, reports mostly of a progress nature from the several subcommittees were presented. In connection with industrial surveys, a valuable feature of the refractories manual, several are being prepared covering new topics as well as revision of old. Surveys are in progress covering lead, copper, coke ovens, flat glass, blast furnace and frit smelter and at the meeting surveys were suggested covering paper, zinc, reheating furnaces, and other processes.

The research subcommittee has prepared an extensive list of 22 research problems concerning the testing of refractories. These have been submitted to the Administrative Committee on Research and the members were urged to offer any available solutions for consideration. The several sections of the subcommittee on tests reported progress generally. It is now considered that the rates of loading, particularly in the low-strength field, are too high and will be reviewed along with those applying to castables and type I fire brick which need revision. A uniform rate of loading is favored using the single point loading method only. In the study of PCE furnaces, six standard samples have been processed into cones which together with PCE cones and cone plaque material will be run in several furnaces in member laboratories. It will be their objective to establish data covering many types of furnaces such as the Denver (natural gas), propane, electrical resistance and oxyacetylene types. Two analytical procedures are being prepared by the section on analysis to cover titanium oxide de-

termination and ignition loss of magnesia refractories. A sonic method for the determination of disintegration is being given attention as a possible criterion by the section on carbon monoxide disintegration. The need for a method of test for measuring heat transfer in castables and plastic refractories was expressed and it is planned to secure data as a start on a heat transfer method.

The only recommendation presented was in the form of a suggested change to the Specifications for Castable Refractories for Boiler Furnaces and Incinerators (C 213-47 T). This change, if approved, will alter the present rate of application of load when using the hydraulic machine to 500 lb. per min. in the modulus of rupture tests. The need was expressed for definitions to cover the terms "mullite" and "sillimanite." In this connection the Subcommittee on Classifications was instructed to consider also "mullite brick". A very comprehensive report was presented by the special refractories subcommittee on the need for establishing a basis for consideration of the refractories which shall be included in this "special" category. In carbon refractories, a new field of coverage by the committee, preliminary work has been started. Two groups of materials comprise this field: namely, carbon refractories which are carbon or essentially carbon and carbon-silica refractories. Both will be studied in connection with established test procedures.

It was agreed that the next meeting of the committee be held during the Spring, 1950, meeting of the Society in Pittsburgh.

**Committee C-21** on Ceramic Whitewares held during the evening its first full meeting since its organization. After a report by Secretary Geller, the applications of additional members were approved by the Executive Subcommittee and brief reports were presented by each subcommittee chairman. A favorable letter ballot was announced on the adoption of by-laws for the committee, and these are now in effect. The subcommittee on nomenclature had held several meetings during the year and the hope was expressed by Chairman Watts that

agreement will be reached before the next meeting on a number of basic terms in the whitewares field, as, for example, china, porcelain, earthenware, vitreous, semi-vitreous, and other similar terms. Sections of the subcommittee on tests and specifications are in varying stages of development and progress. The section on raw materials has inaugurated work on feldspar. Correspondence is being exchanged within the section on processing controls to establish initially what tests are now being used. In considering fundamental properties of products—water absorption, porosity and specific gravity—test methods are receiving attention.

The subcommittee on research, now fully organized, has already completed some of its initial projects. It is planned to coordinate the research with that of several universities whereby projects may be set up for graduate thesis work. It is expected that the next meeting of the entire committee will be held during the 1950 Spring Meeting of the Society in Pittsburgh with plans to include subcommittee meetings.

### Meeting of Committee C-16 on Thermal Insulating Materials

In colonial Williamsburg, Va., Committee C-16 on Thermal Insulating Materials held meetings to discuss one of the more modern materials, thermal insulation. A three-day meeting consisting of all subcommittees and a main meeting was held at the Williamsburg Inn on September 26-28 with 42 members present.

A few of the highlights of this group of meetings are given. A meeting of the Planning Subcommittee resolved several matters of policy, such as emphasizing the necessity for a significance of test statement to be developed concurrently with the test method itself. The provision was made that the matter of emphasis is to be cleared through the new Subcommittee T-X on Significance of Tests. The need for uniformity of tests was stressed, and each subcommittee will review all existing A.S.T.M. methods before developing a new standard and attempt to coordinate standards with those of other organizations, especially that of the Federal Specification Board.



Among the significant items under discussion or acted upon by the subcommittees were those briefly noted as follows. With the publication by the Society of a new tentative method for evaluating the properties of building boards, submitted by Committee D-7, there appears to be some confusion in industry as to its usage as compared with the earlier A.S.T.M. Methods of Testing Structural Insulating Board Made from Vegetable Fibers (C 209) developed by Committee C-16. The Subcommittee on Insulating Board has recommended that the terms "insulating board" and "building board" be clarified, as well as of such terms as conductivity and density. The subcommittee feels that insulating board should be tested according to Method C 209, and if more detailed structural tests are needed for research, perhaps then the new method developed by Committee D-7 should be considered. Further revisions toward the correlation between the Forest Products Laboratories test methods and those referred to in Federal Specification SS-A-118 were recommended.

A proposed method for determining the adhesion to steel of insulating cement is being used by four cooperating laboratories in order to establish data on this method. Three new specifications were authorized for distribution and letter ballot to the Subcommittee on Blanket Insulation. These cover mineral wool blanket insulation (metal mesh covered), mineral wool industrial batt insulation, and mineral wool felt insulation. The development of a tentative test method for fire resistance of thermal insulating materials was deferred pending writing of a significance of test statement which would govern the scope of such a method. A proposed method for the measurement of density for loose fill insulations is now ready for submittal to letter ballot of the subcommittee. The durability of loose-fill insulations and a correlation of field observations will be the next item of activity by the subcommittee. A revision is contemplated of the Tentative Method of Test for Thermal Conductance.

A symposium is planned for the 1950 Spring or Annual Meeting to be sponsored chiefly by the Subcommittee on Special Thermal Properties. The emphasis of this symposium will be on such subjects as the evaluation of the effect of temperature on thermal insulating materials. One paper is proposed on the subject of measuring specific heat. Work by the Subcommittee on Dimensional Standards in securing field data has been retarded considerably by shutdowns in certain plants. However, it is expected that the survey on field clear-

ances will be completed by the time of the next meeting.

It was voted to change the title of the Subcommittee on Vapor Barriers to that of Vapor Transmission, and likewise to change the title of the new tentative now being processed to that of Water Vapor Transmission of Thermal Insulating and Correlated Materials. These changes will, therefore, enable this method to include materials other than vapor barriers.

The next meeting of the committee will be held in Pittsburgh simultaneously with the Society's 1950 Spring Meeting

### Committee C-19 on Structural Sandwich Constructions

A MEETING of Committee C-19 on Structural Sandwich Construction was held at Society Headquarters on September 29 and 30. The Subcommittee on Mechanical Properties of Basic Materials submitted for discussion a compilation of physical tests on core material accumulated from various sources. These were divided into five groupings as follows: (1) thermal properties, (2) water absorption, (3) dimensional stability, (4) bondability, and (5) apparent and absolute density. Each of these groupings was discussed and it was decided that proposed tentative methods embodying the best points of the various methods submitted should be drafted for each group.

There was also discussion of proposed tentative methods of testing sandwich construction in shear, edgewise; also in shear, flatwise plane. These proposed tentative methods will be revised in line with the recommendations of the subcommittee and will be subsequently sent out to letter ballot of the subcommittee.

The Subcommittee on Permanence, Durability and Simulated Service discussed a proposal to expose various types of sandwich constructions at the A.S.T.M. test sites. Their plan, although in the initial stages, anticipates exposure of at least 50 panels, 12 by 12 in. minimum size, at each site.

The Advisory Subcommittee recommended that the By-laws should be amended (a) to provide an additional officer to be known as a membership secretary and (b) to make a provision that only one meeting a year need be held. The present By-laws state that there shall be two.

It was announced that plans were complete for the meeting of Committee C-19 during the Pacific Area National Meeting in San Francisco on October

14, with Vice-Chairman Reid in charge of a program of papers to be presented on subjects dealing with sandwich construction.

### Committee C-1 Approves Addition to Portland Cement

It is announced by Committee C-1 on Cement that the material known as "N-TAIR" has been declared acceptable as an addition to the cements covered in tentative A.S.T.M. specifications C 175 and C 205.

This recognition is in accordance with the policy of the committee and the Society in indicating the acceptability of additions to portland cement following the completion of suitable tests and review of existing data. The present specifications mentioned above will appear soon, revised by the addition of an appropriate footnote which is essentially as follows:

A material known commercially as N-TAIR, which is manufactured by Newport Industries, Inc., and consists substantially of a sodium resinate produced from pine wood stumps from which the bulk of the petroleum naphtha-soluble resin acids has been removed, is acceptable. The resin, if regenerated from the sap, has an acid number not less than 125. The N-TAIR has been stabilized so that it will remain homogeneous and completely soluble at all working concentrations.

### Treatise on Powder Metallurgy

VOLUME I, Technology of Metal Powders and Their Products is an encyclopedic treatment of powder metallurgy by Claus G. Goetzel, Ph.D., Vice-President and Director of Research, Sintercast Corporation of America, N. Y., and presents 20 chapters of principles, history, theory, experiment, technology, properties, production, and application.

Through powder metallurgy's techniques, whole new families of products such as the cemented carbides and a host of other special-property metal parts and materials, are being produced now that could not be produced by other means ten years ago.

Following the orientation, there are chapters which describe methods of powder production, characteristics, properties and standards of available powders, methods of testing powders, powder conditioning, and the function of addition agents.

Among numerous topics covered, the underlying principles of powder compaction, methods of fabrications and equipment used, and the intering process, are given thorough treatment. Three final chapters relate to the operations that follow sintering.

Mr. Goetzel's first volume (of three), published by Interscience Publishers, Inc., New York, N. Y., is made up of 806 pages with 300 illustrations and 82 tables, and sells for \$15.

## Active Standardization Program Continues in Petroleum Committee's Work

### Extensive Reorganization Being Completed

IN THE September issue of the ASTM BULLETIN, pages 20 to 30, an annual "round-up" of important standardization activities was given. Now, based on a work program submitted by the officers of Committee D-2 on Petroleum Products and Lubricants and presented in the interests of the membership broadly, an important addition to the "round-up" follows.

Committee D-2 on Petroleum Products and Lubricants is just now completing its subcommittee reorganization which has been under way for the past two years. This reorganization has not only caused better distribution of the responsibility for the research and standardization programs under way but has resulted in the formation of several new groups.

The work of the committee is proceeding at a very satisfactory rate. It involves a large number of independent projects being carried on by individual sections of Technical Committees or Research Divisions. The more important of these projects are outlined in the following paragraphs.

Technical Committee A on Gasoline is continuing its study of the specification for gasoline in order to make sure that this specification is in line with current commercial practice. The accent at this time is on the vapor pressure and octane limits as they apply to current requirements. A section of the committee is currently studying information which is available concerning the effect of motor fuels on engine varnish and sludge.

The most active project under the sponsorship of Technical Committee B on Lubricating Oils continues to be that on the study of crankcase oil classification systems. A review is currently being made of a questionnaire which was distributed to the membership of Committee D-2 and the SAE Fuels and Lubricants Technical Committee during the latter part of June. Another program which has just been initiated within this committee is a study which will lead to the development of more satisfactory test methods for the measurement of the dilution of crankcase oils by gasoline and diesel fuels.

Technical Committee C on Turbine Oils has a number of active projects under way. A new section on Compatibility Testing of Turbine Oils, of Technical Committee C is being formed.

Technical Committee F on Diesel Fuels is sponsoring a symposium at the time of the San Francisco meeting of the Society entitled "The Effect of Fuel on Diesel Engine Deposits." This symposium is the outgrowth of a discussion at the February meeting of Technical Committee F and will assist the committee in determining those diesel engine problems which most require its attention.

Technical Committee G on Lubricating

Grease has undergone a reorganization of its own as the result of which it now comprises 14 active subsections each of which is responsible for a single test method or for the work which will lead to the development of a test method. Technical Committee G plans to recommend the adoption of Method D 942, Test for Oxygen Stability of Lubricating Greases by the Oxygen Bomb Method, and Method D 972, Test for Evaporation Loss of Lubricating Greases and Oils as standard during the course of the next year.

Two sections of Technical Committee H on Light Hydrocarbons are active at present. The first, on Chemical Utilization, plans to take up improvements in the Rubber Reserve butadiene methods which heretofore have been accepted with editorial revisions only. The second, on Liquefied Petroleum Gas, has initiated considerable work on methods for the determination of vapor pressure, specific gravity, sulfur, and corrosion.

Sections of Technical Committee K on Cutting Fluids are working on an evaluation of emulsion stability tests, the development of a method of test for active sulfur in cutting fluids by means of the copper powder method, and the development of a test procedure for running tool-life tests on a lathe.

The Special Subcommittee on Extreme Pressure Properties Measurement is currently carrying out cooperative tests by the Timken Test using a single procedure, the details of which are being formulated.

Research Division I on Combustion Characteristics is showing especial interest at present in a special project being carried on at the National Bureau of Standards at the Division's instigation for the determination of the effect of barometric pressure on the Knock Test methods. It is undertaking a study of the F-21 aviation test method, and it hopes to develop a micro-method of test which will be used primarily in evaluating the knock characteristics of primary reference fuels.

Division II on Measuring and Sampling is just completing the text of the first volume of an "ASTM Manual on Measuring and Sampling Petroleum and Its Products." Clearance has already been obtained for publication of the book, which will contain new methods covering the measuring, gaging, and temperature measurement of petroleum products. Division II is also actively engaged in the ASTM-IP project for the preparation of a comprehensive oil volume measurement tables.

The newly organized Research Division III on Elemental Analysis sponsored a Symposium on Modern Instrumental and Chemical Methods for the Determination of Metal in Petroleum Products at the San Francisco meeting of the Society.

Research Division IV on Hydrocarbon Analysis is completing methods of test for

the analysis of diesel fuels, the infrared analysis of 60-40 blends of iso-octane and normal heptane, the infrared analysis of C<sub>4</sub> fractions and the ultraviolet determination of butadiene.

Research Division V on the Analysis of Fuels which has just been organized is interested at present in such diverse subjects as the development of a method of test for gum in jet fuels and the improvement of Method D 96, Test for Water and Sediment in Petroleum Products by Means of Centrifuge.

Section C on Carbon Residue of Research Division VI on Analysis of Lubricants is continuing its active study into the factors governing the reproducibility of Method D 524, Test for Carbon Residue in Petroleum Products (Ramsbottom Carbon Residue).

Research Division VII on Flow Properties has recently been organized for the purpose of centralizing the work being carried on in Committee D-2 on viscosity and similar properties. It is currently interested in the revision of Method D 445, Test for Kinematic Viscosity, and in the study of the pour stability characteristics of winter grade motor oils.

Section E on Vacuum Distillation of Research Division VIII on Volatility is planning to publish the preliminary text of a method of test for the vacuum distillation of petroleum fractions during the coming year.

Research Division IX on Color is continuing to investigate the limits of precision of Method D 156, Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer, and to carry out chromatic measurements for the standardization of the glass color standards used in Method D 155, Test for Color of Lubricating Oils and Petroleum by Means of the ASTM Colorimeter.

Research Division X on Corrosion Tests is looking forward to further refinements of D 130-49 T, the tentative revision of the Standard Method of Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline. Among these will be the definition of a series of standard color strips for comparison purposes.

Subcommittee I on Pharmaceutical Tests is carrying out a program for the improvement of the reproducibility of Method D 937, Test for Penetration of Petrolatum.

Subcommittee XXIV on Petroleum Sulfonates is planning to investigate the use of hydrocarbons other than petroleum ether for de-oiling purposes and to develop repeatability and reproducibility limits for Method D 855, Analysis of Petroleum Sulfonates.

Coordinating Divisions within the D-2 organization are developing the information necessary for the preparation of a new edition of the booklet on the significance of the test methods under D-2 jurisdiction, investigating the expanded use of statistical methods in connection with its programs, and carrying on other similar projects.



# **Treasurer J. K. Rittenhouse, at A.S.T.M. for 40 Years**

AT THE September meeting of the Board of Directors an appropriate resolution was offered, recognizing and expressing appreciation for the 40 years of continuous service on the Headquarters Staff by the current Treasurer, John K. Rittenhouse.

The resolution reads in part:

"...the Board of Directors is pleased to note that on October 9 of this year he will complete forty years of continuous service on the staff of the Society. He was the first full-time employee, and in those early days of our history he not only kept the Society's books but also recorded the minutes of meetings of the Executive Committee, then the governing body of the Society, and performed other duties that as the Society grew have been taken over by others. He was subsequently appointed Assistant Treasurer, and since 1945 has been Treasurer of the Society.

"He has served the Society faithfully and well over this long-extended period, and the Board wishes to record and express its appreciation to Mr. Rittenhouse for his services and especially to recognize and offer to him its felicitations on the completion of forty years' affiliation with the staff of the Society."

## **Addition to Technical and Editorial Staff**

TO AID in handling the steadily increasing technical and editorial work, there has been recently appointed to the staff Mr. William F. Black, Jr. Mr. Black graduated in June, 1949, from Drexel Institute of Technology with the degree of B.S. in Chemical Engineering, after serving for five years in the U. S. Army, Medical Administrative Corps, which he left in 1946 with the rank of captain. Prior to entering the Army he had worked with Sharp and Dohme and the Du Pont Company. Mr. Black is reporting to the Technical Secretary and Editor and will be engaged in editorial work, including that of the ASTM BULLETIN, and in working with some of the A.S.T.M. Committees.

## **Strength of Materials**

By Joseph Marin

ONE of the main features of this book by Joseph Marin, Professor of Engineering Mechanics, The Pennsylvania State College, and long-time active A.S.T.M. member, is the treatment of mechanical properties of materials under various types of static stresses coordinated with the analysis of stresses and design of simple machine and structural members. The correlation between stress analysis, mechanical properties, and design is made possible by a discussion of mechanical properties at the end of each chapter. In this way, the student is better prepared to proceed with the study of machine, structural, and aircraft design.

The arrangement of the material in four parts—Simple Stresses, Combined Stresses, Statically Indeterminate Stresses, and Special Topics—is believed to give a more understandable treatment of the subject for undergraduate students. The contents of this book according to chapter under each part will present a view of the material covered as follows.

*Part I Simple Stresses.*—Chapter I Members Subjected to Simple Tension and Compression, Chapter II Members Subjected to Shear and Torsion, Chapter III Members Subjected to Bending—Beams, Chapter IV Members Subjected to Axial and Bending Loads—Short Members, Chapter V Members Subjected to Axial and Bending Loads—Long Members or Columns.

*Part II Combined Stresses.*—Chapter VI Members Subjected to Combined Stresses—Determination of Stresses, Chapter VII Members Subjected to Combined Stresses—Theories of Failure and Design.

*Part III Statically Indeterminate Stresses.*—Chapter VIII Deformation Methods of Analysis, Chapter IX Energy Methods of Analysis.

*Part IV Special Topics.*—Chapter X Riveted and Welded Joints, Chapter XI Special Problems on Bending of Beams, Chapter XII Members Subjected to Fatigue and Impact Loads.

*Appendix I.*—Centroids and Moments of Inertia of Plane Areas.

*Appendix II.*—Tables of Physical Properties and Design Stresses for Common Engineering Materials.

This 464-page publication may be obtained at a cost of \$4.75 per copy from The Macmillan Co., 60 Fifth Ave., New York, N. Y.

# **Story of a War Contractor**



This "story" recently came to the attention of a staff member, and is shown here even though a number of our members and readers may have noted it before. We are not certain of the original source (somehow it must get back to a zoo), but it appeals to us as one of the best and most trenchant stories, "physiognomonically" speaking, we have seen.

## New Members to September 21, 1949

The following 44 members were elected from August 18, 1949, to September 21, 1949, making the total membership 6622.

Names are arranged alphabetically—company members first, then individuals.

### Chicago District

CLEARING MACHINE CORP., E. K. Johansen, Vice-President, 6499 W. Sixty-fifth St., Chicago 38, Ill.  
ECONOMICS LABORATORY, INC., John L. Wilson, Director of Research, 914 Guardian Bldg., St. Paul 1, Minn.  
COLBURN, WILLIAM, President, Colburn Laboratories, Inc., 431 S. Dearborn St., Chicago 5, Ill.  
KUEMMERLING, OTTO, Technical Superintendent, Wausau Paper Mills Co., Brokaw, Wis.  
RUEMMER, W. P., Plant Manager, The Eagle-Picher Lead Co., Box 540, East Chicago, Ind.  
SVOREN, LOUIS JAMES, Draftsman, J. I. Case Co., Tractor Works, Racine, Wis. For mail: 943 Wilson St., Racine, Wis. [J]\*

### Cleveland District

CAHN, HAROLD L., Chemist, Interchemical Corp., Murphy Paint Div., Wooster, Ohio.  
HARDGROVE, RALPH M., Director of Product Development, Babcock & Wilcox Co., Research Lab., Alliance, Ohio.  
HOLTON, ROBERT J., Chief Inspector, Tinnerman Products, Inc., 2038 Fulton Rd., Cleveland, Ohio.

### Detroit District

Fry, G. D., Chief Engineer, Schultz Die Casting Company of Canada, Ltd., Wallaceburg, Ont., Canada. For mail: 332 Camp St., Wallaceburg, Ont., Canada.  
MACKAY, H. W., Superintendent, Product Engineering, Briggs Manufacturing Co., Eight Mile Plant, Detroit, Mich. For mail: 10731 Wayburn, Detroit 24, Mich.  
OVEN, GERALD E., Paint Chemist, Kaiser-Frazer Corp., Paint Dept., Willow Run, Mich.

### New England District

KOERNER, GORDON L., Factory Manager, Rhode Island Tool Co., 148 W. River St., Providence 1, R. I.  
THOMAS, HENRY E., Assistant Professor,

Lowell Textile Inst., Lowell, Mass. For mail: 779 Westford St., Lowell, Mass.  
ZUSSMAN, H. W., Vice-President, Alrose Chemical Co., Box 1294, Providence 1, R. I.

### New York District

NOPCO CHEMICAL CO., G. D. Davis, Vice-President, First and Essex Sts., Harrison, N. J.  
HARRIS, ALFRED C., Metallurgist, Kraeuter and Co., Inc., 585 Eighteenth Ave., Newark 3, N. J.

### Northern California District

CONRAD, CHARLES S., Director, Metal Sales, Tay-Holbrook, Inc., 165 Eighth St., San Francisco, Calif.  
SAN LEANDRO, CITY OF, Charles P. Martin, Public Works Director, City Hall, San Leandro, Calif.

### Ohio Valley District

(In Course of Organization)

ILLING, ARNO M., Manager, Development Dept., Cambridge Tile Manufacturing Co., Box 71, Cincinnati 15, Ohio.  
SHEPARD, CHARLES H., Engineer, Ohio State Highway Testing and Research Lab., Ohio State University Campus, Columbus 10, Ohio.

### Philadelphia District

LONDON, GEORGE K., Technical Sales and Service, Standard Paper Manufacturing Co., Richmond, Va. For mail: 3100 Pennsylvania Ave., Wilmington, Del.  
MUMBERG, FRITZ, Glassblower, Ace Thermometer and Instrument Co., Rockledge, Pa. For mail: 361 Gilham St., Philadelphia 11, Pa.  
VILLANOVA COLLEGE, MATERIALS TESTING DEPT., Joseph C. Greyson, Associate Professor of Mechanical Engineering, Villanova, Pa.

### Pittsburgh District

SMITH, CHARLES A., JR., Superintendent, Oil City Sand and Gravel Co., Mounted Rt. 15, Innis St., Ext., Oil City, Pa.

### St. Louis District

ANDERSON, ROLLAND L., Director of Research and Development, Chicago & Southern Airlines, Municipal Airport, Memphis, Tenn. For mail: 389 Holmes Circle, Memphis, Tenn.

### Washington (D. C.) District

INSTITUTE OF COOKING AND HEATING

APPLIANCE MANUFACTURERS, Samuel Dunkel, Managing Director, Shoreham Hotel, Washington 8, D. C.  
O'HARA, DONALD C., Attorney, National Petroleum Assn., 958 Munsey Bldg., Washington 4, D. C.  
SITTLER, HOWARD L., Metallurgist, Arcrode Corp., Sparrows Point 19, Md. For mail: 624 Round Oak Rd., Towson 4, Md.

### Western New York-Ontario District

SCHLEICHER, MARTIN E., Technical Director, McDougall-Butler Co., Inc., 6 Evans St., Buffalo 5, N. Y.

### U. S. and Possessions

BRISTOW, JOHN, General Manager, King Bros., Inc., 3500 S. E. Seventeenth St., Portland, Ore. For mail: 11848 S. E. Rhone St., Portland, Ore.  
CHA, S. H., Chief Chemist, Canec Div., Flintkote Co., Box 647, Hilo, Hawaii.  
DEVANEY, L. W., Co-owner, Waco Chemical Testing Lab., 2914A S. Third, Waco, Tex. For mail: Box B. U. Station 396, Waco, Tex.  
JONES, WILLIAM WORTH, Chief Chemist, Plymouth Oil Co., Sinton, Tex. [J]  
PUTNAM, G. C., Technical Superintendent, E. I. du Pont de Nemours and Co., Inc., Box 2027, Orange, Tex.  
RAY, CARL J., President, Commercial Testing Labs., 519 Lipan, Denver 4, Colo.  
TAMPA, CITY OF, Roy K. Van Camp, Superintendent of Public Works, Fifth Floor City Hall, Tampa 2, Fla.

### Other than U. S. Possessions

SOCIETE ANONYME JOHN COCKERILL, Secretariat General, Seraing, Belgium.  
BIBLIOTHEK DER TECHNISCHEN HOCHSCHULE, P. Gehring, Director, Keplerstr. 10, Stuttgart N, Germany.  
DATE, MADHUSUDAN MORESHWAR, Chief Chemist, Asbestos Cement, Ltd., Mulund, Bombay, India. For mail: Pathare House, Bhawani Shankar Rd., Dadar, Bombay 14, India.  
GOODBRAND, A. N., Goodbrand and Co., Ltd., Britannia Foundry, Stalybridge, Cheshire, England.  
HARTEN, KARL PETER, Geschäftsführer, Verein Deutscher Eisenhüttenleute, Aug-Thyssen-Str. 1, Dusseldorf, Germany.  
MEHTA, R. K., Technical Director, Kamani Metals and Alloys, Ltd., Agra Rd., Kurla, Bombay, India.  
WILLEY, GORDON E., Chief Metallurgist, Algoma Steel Corp., Ltd., Sault Ste. Marie, Ont., Canada.

\* [J] denotes Junior Member.

## PERSONALS • • •

News items concerning the activities of our members will be welcomed for inclusion in the column.

NOTE—These "Personals" are arranged in order of alphabetical sequence of the names. Frequently two or more members may be referred to in the same note, in which case the first one named is used as a key letter. It is believed that this arrangement will facilitate reference to the news about members.

Edgar Collins Bain, Vice-President in Charge of Research and Technology, Carnegie-Illinois Steel Corp., Pittsburgh, Pa., and noted authority on the fabrication and properties of metals, has been awarded the John Price Wetherill Medal of the Franklin Institute of the State of Pennsylvania. The award was given in recognition of Dr. Bain's investigation into the structure of metallic alloys and his original work in application of the iso-

thermal method of studying the rates of the transformations in steel which are responsible for its hardening.

Ladislav Boor, of Bethesda, Md., is now Technologist at the Philadelphia Quartermaster Depot, Chemical & Plastics Laboratory, Philadelphia, Pa.

Hyman Bornstein, formerly Director of Laboratories, Deere and Co., Moline, Ill., is now Manager, Materials Engineering Dept. Mr. Bornstein, very active in

A.S.T.M. work for many years, has as his most recent contribution to the work the development of the Session on Cast Iron being held as a technical feature of the Pacific Area National Meeting in San Francisco.

James A. Broadston, formerly Armament Design Engineer for North American Aviation, Inc., Los Angeles, Calif., has been advanced to a new position as Propulsion Test Group Leader of North American's Aerophysics Field Laboratory for the research and development testing of large rocket power plants for guided missiles. Mr. Broadston also serves as Chief Engineer of Surface Checking Gage Co., Hollywood. A new third edition of his text "Control of Surface Quality" has just been published that brings up to date information on certain new ASA "Surf-Chek" Roughness Standards for designating the roughness of machined surfaces.

L. H. Cohan, formerly Director, Witon Chemical Co., New York City, is now Chemist, Hanline Bros., Baltimore, Md.



**Carl G. Crawford**, Vice-President, American Creosoting Co., Louisville, Ky., has been elected to Honorary Membership in the American Wood-Preservers' Association, in recognition of his many contributions to the advancement of the art of wood preservation in the United States.

**Fred W. Gottschalk**, Technical Director, American Lumber & Treating Co., Chicago, Ill., has been elected first Vice-President of the American Wood-Preservers' Association. During the past year Mr. Gottschalk as second Vice-President directed a campaign which resulted in an 18 per cent membership increase, the number of members in the Association now totaling 1448.

**M. A. Teixeira DeCastro**, formerly Technical Director, Companhia de Cimento, Portland Paraiso, São Paulo, Brazil, is now in business for himself as engineering consultant in São Paulo.

**Philip J. Elving** has returned to The Pennsylvania State College as Professor of Analytical Chemistry. He left Penn State in 1939 to go to Purdue University where he served on the faculty as Associate Professor of Chemistry until this year, except for a period of four years during the war when he was with Publicker Industries, Inc., of Philadelphia, as Assistant Director of Chemical Research.

**F. Malcolm Farmer**, formerly Vice-President, has been elected President of Electrical Testing Laboratories, New York. Joining ETL in 1903, he became Chief Engineer in 1911 and Vice-President in 1929. Mr. Farmer is a Past-President and Honorary Member of A.S.T.M., also has served as President of A.I.E.E., A.W.S. and of United Engineering Trustees.

**Arno C. Fieldner**, Chief of the Fuels and Explosives Division of the United States Bureau of Mines, was honored with the Distinguished Service Award and Gold Medal by the Department of the Interior, on August 30, in recognition of his notable contributions to fuel technology and of more than 40 years of exemplary public service.

**A. T. Goldbeck**, Engineering Director, National Crushed Stone Assn., Washing-

ton, D. C., and long-time A.S.T.M. member, has this year become a Life Member of the American Society of Civil Engineers. Affiliated with A.S.T.M. since 1910, Dr. Goldbeck has rendered active service through the years on various technical committees, especially C-9 on Concrete of which he was Chairman and Secretary, respectively, for four-year periods. He also served on the Executive Committee and is currently a member of the Washington District Council.

**Martin H. Gurley, Jr.**, has been appointed Research Director for the American Textile Co., Inc., Pawtucket, R. I. He was previously in charge of textile and paper research and development at the Esselen Research Corp.

**J. Earl Harrington**, for many years with the Bureau of Engineering of the City of Chicago, and later during the war consultant and engineering coordinator for the Quartermaster General, and engineer with the Explosives Branch of the Ordnance Department, has been named Executive Secretary of the Western Society of Engineers.

**Don R. Learned**, formerly with the U.S.N.R., is now Facility Analyst at Ford Motor Co., Detroit, Mich.

**Harry Majors, Jr.**, has been named Director, Engineering Experiment Station, University of Alabama. He was formerly on the faculty at the Massachusetts Institute of Technology.

**Herman Mark**, Director of the Institute of Polymer Research at the Polytechnic Institute of Brooklyn, has been invited to deliver a general lecture on plastics at the International Congress of Plastics in Turin, Italy.

**P. B. Mayfield** is now Technical Consultant, The Barrett Div., Allied Chemical & Dye Corp., New York City. He was formerly Manager, Research & Development, International Creosoting & Construction Co., Galveston, Tex.

**J. Strother Miller**, Asphalt Technologist, has moved to his new home at 63 Bernhardt Drive, Snyder, Buffalo 21, N. Y., from his former temporary address on Campus Drive, Buffalo. Affiliated with

A.S.T.M. since 1903, Mr. Miller was made an Honorary Member in 1946.

**LeRoy L. Peterson** has accepted a position as Chemical Engineer, Kimberly-Clark Corp., Memphis, Tenn. He was previously Chemical Research Engineer, Michigan State Highway Dept., Lansing, Mich.

**R. E. P. Shearer**, previously Technical Adviser, The New Hindustan Cements, Ltd., Bombay, India, is now a member of the firm, Shearer, Foges & Partners (Consulting Engineers), of the same city.

**Harold M. Smith**, Principal Petroleum Chemist at the Bureau of Mines Petroleum Experiment Station, Bartlesville, Okla., was honored recently by the highest award that the Department of the Interior can bestow upon one of its employees. The gold medal and accompanying citation with which he was presented were in recognition of his 26 years of service with the Bureau of Mines, and for his contributions to petroleum chemistry. The citation reads in part as follows: "For an outstanding scientific career, with technical contributions to knowledge recognized wherever the chemistry of petroleum and natural gas is studied, and for contributions to the allied effort through careful technical planning and wise supervision of research conducted to meet war requirements for aviation gasoline and other petroleum munitions." A member of A.S.T.M. for many years, Mr. Smith has been very active in the work of Committee D-2 on Petroleum Products and many of its subgroups.

**Structural Clay Products Institute** has recently announced its new address as 1520 18th St., N.W., Washington 6, D. C. The Institute's new quarters are larger and will permit expansion of phases of the organization's work.

**Louis A. Weinland** has been named Assistant Professor of Chemistry, New York State College of Ceramics, Alfred University, Alfred, N. Y. He was formerly Associate Professor of Chemistry, Champlain College, Plattsburg, N. Y.

## NECROLOGY

**LEROY S. CONVERSE**, Director, Standards Dept., American Viscose Corp., Marcus Hook, Pa. (July 6, 1949). Mr. Converse died suddenly, collapsing at the wheel of his car. Prior to his death he had represented his company's Sustaining Membership for some time, also had served on Committee D-13 on Textile Materials since 1941, being Secretary of its Subcommittee on Tire Cord and Fabrics, and was a consulting member of Committee E-9 on Fatigue.

**H. B. GREENSTED**, Metallurgical Consultant, Algoma Steel Corp., Ltd., Sault Ste. Marie, Ontario, Canada (September 1, 1949). Member since 1935.

**WILLIAM J. JEFFRIES**, Metallurgical Consultant, Cape May, N. J. (September 1, 1949). A member since 1935, Mr. Jef-

fries was very widely known in A.S.T.M., in part because of his long-time service with the U. S. Navy Department. For many years he served in the Bureau of Engineering and represented that Bureau in many of the A.S.T.M. activities. Later he was technical head of the Philadelphia Ordnance District, and subsequently had been in consulting practice. He has served on a number of A.S.T.M. technical committees including Committee A-1 on Steel and Committee A-5 on Corrosion of Iron and Steel, and was also on the Philadelphia District Council.

**HENRY M. MILBURN**, Senior Engineer of Tests, Bureau of Public Roads, Department of Commerce, Washington, D. C. (September 20, 1949). A member of the Society since 1917, Mr. Milburn was active in the work of Committee D-4 on Road and Paving Materials for many

years, serving as chairman of several of its subcommittees and as representative of D-4 on Committee E-1 on Methods of Testing. He had been with the Bureau of Public Roads for the last 31 years. Mr. Milburn died at his home in Kensington, Md., of a heart attack at the age of 69.

**JOHN JAY SHANK**, Owner and Director, The Wayne Laboratories, Waynesboro, Pa. (September 16, 1949). Representative of The Wayne Laboratories' membership since 1932, and member of Committee D-8 on Bituminous Waterproofing and Roofing Materials for many years.

## Notes on Laboratory Supplies

### Catalogs and Literature; Notes on New or Improved Apparatus

This information is based on literature and statements from apparatus manufacturers and laboratory supply houses.

#### Catalogs and Literature

**Buehler Ltd.**, 165 W. Wacker Drive, Chicago 1, Ill. A most interesting and practical reference booklet for use when ordering polishing cloth and abrasives for metallographic work has been issued. "Polishing Cloth and Abrasives," includes twenty pages and small sample swatches of the material described, for more practical inspection before ordering. The foreword in the booklet sets forth very ably its purpose and general nature and is quoted in part:

"Polishing Cloth and Abrasives" has been prepared for our friends in the metallurgical laboratories and purchasing departments to assist them in the proper selection of laboratory supplies as well as to help the buyer in the correct specification of the material selected..."

**Scientific Glass Apparatus Co., Inc.**, Bloomfield, N. J. This is the latest issue, Number Eight, of "What's New for the Laboratory." It illustrates and describes many instruments and pieces of equipment, such as Gyco Heating Jackets, Abbe-56 Refractometer, Funnels and Graduates made of Durable Plastic, Hygrometers, Imported Agate Mortars, Pyrometers, Thermometers, Trip Balances, and others. 16 pages. Summer, 1949, edition.

**Wilkens-Anderson Co.**, 4525 W. Division St., Chicago 51, Ill. "Waco Catalyst," Volume VIII, announces Wilkens-Anderson Co.'s new address (noted above). It illustrates and describes a number of instruments including Ainsworth Balances and Weights, Triple Beam Balance, Walter Baths, Buffer Tablets, Burettes, Oil Refining Cup, Boekel Pipette Washer, Precision Shaker, Spectrophotometers, Stirring Paddles, pH Meters, pH Test Papers, Timers and Stopclocks, Titration Assembly, Midget Pump, and many others. Includes an index of items listed. 32 pages, 8½ by 11 in.

**Bausch & Lomb Optical Co.**, Rochester 2, N. Y. Catalog E-30, entitled "Bausch & Lomb Optical Glass," is the first optical glass catalog issued by the company since the start of World War II and contains much information which will be of interest to engineers and designers of instruments utilizing fine glass optical elements. The catalog tells two separate, but related stories—one, in pictures, of the manufacture of optical glass in the Bausch & Lomb Glass Plant—the other, the technical specifications of the available B&L optical glass. Sections of the catalog cover Optical, Physical, and Chemical Properties, and a section covering General Data. 24 pages, 8½ by 11 in., illustrated.

**Laboratory Equipment Corp.**, St. Joseph, Mich. "LECO Zircon Refractory Ware Catalog" illustrates and describes combustion tubes, stock size tubes, combustion tube liners, combustion boats and shields, ignition ware, high-and low-temperature, melting crucibles, and refractory plate, flame ring, etc. A price list is included. 22 pages, 8½ by 11 in., illustrated.

#### Instrument Notes

**Baldwin-Hunter Spring Tester**—The Baldwin Locomotive Works, Philadelphia 42, Pa. This machine was designed for high production inspection and testing of any type of small spring (coil, flat, special design, or spring assembly) within the limits of 12 in. in length and 5 lb. of load. It is especially useful for springs made of wire sizes from 0.005 in. to 0.030 in. in diameter. The equipment is sensitive to load changes of less than 50 mg. and has a weighing accuracy within  $\pm 0.10$  per cent.

**SR-4 Strain Indicator**—The Baldwin Locomotive Works. A new self-balancing, dial-type strain indicator for use in stress analysis with SR-4 bonded resistance wire strain gages. The automatic balancing feature simplifies strain measurement by giving quick indications of strains on a dial scale calibrated to read directly in micro-inches per inch for standard gages (gage factors from 1.90 to 2.20). Adjustments for gage factor and for zero setting are provided above the dial inside the case. The indicator can be used either with a single strain gage or with multi-point switching and balancing units for reading many strain points.

**Butyl Rubber Tubing**—Fisher Scientific Co., 717 Forbes St., Pittsburgh, Pa. Formerly obtainable only in large quantity lots, Butyl rubber tubing may now be purchased in quantities convenient for the laboratory. Tubing comes in two diameters—¼ in. and ⅜ in., both with ¼-in. wall thickness. Gas analysis equipment gives accurate results when Butyl rubber tubing is used in place of other types, because of its low permeability to air. It is soft and flexible, and therefore fits snugly over glass fittings.

**Refrigerated Bath for Laboratory Use**—Fisher Scientific Co. This bath obviates the need for make shift arrangements and use of ice in cooling reaction vessels with their contents. It is of particular usefulness in steel or metallurgical laboratories for cooling volumetric test samples, in water and sewage laboratories, and wherever exothermic reactions are under study. The new bath is a copper-lined, steel cabinet with tray adjustable by means of supporting chains over height of 10 in. A sensitive thermostat and control provide low temperatures which can be maintained for 15 to 50 gal. of water at any point from 0 C. to room temperature.

**Spectrogram Comparator M1176**—The Gaertner Scientific Corp., 1201 Wrightwood Ave., Chicago 14, Ill. This Comparator is of the traveling microscope type intended for measuring spectrographic plates and similar records. The instrument reads to 1 micron over a range of 155 mm. The microscope has rack and pinion focusing adjustment and an adjustable draw tube which can be clamped in position. It is furnished with two achromatic objectives and two eyepieces, permitting the use of any magnification from 5 to 40 diameters. A cover for protection against

dust and moisture, and an accessory box containing the extra eyepiece and objective, camel's hair brush, screw driver, and bottle of clock oil are furnished with the instrument.

**Contact Modulated Amplifier**—General Motors Corp., Detroit, Mich. A super-sensitive instrument that can measure the cooling rate of steel during quenching is the so-called Contact Modulated Amplifier which was developed by GM Research during the early part of the war. The research objective was to perfect a device that would measure direct current signals as faint as a few hundredths of a microvolt, a task ordinarily performed by a sensitive galvanometer. The amplifier can be handled virtually like a portable radio and operates almost anywhere with undisturbed stability. Linked with thermocouples attached to a test bar of steel, the amplifier instantly records the cooling rate which always is extremely fast in the initial moment of the quench, and aids in classifying steels according to their hardness or hardenability.

**Type E Electro-Chemograph**—Leeds & Northrup Co., 4934 Stenton Ave., Philadelphia 44, Pa. Designed for rapid, accurate polarographic analyses, this new Chemograph features one-piece console design incorporating a built-in Speedomax Recorder. The instrument is convenient for both research and industrial process control laboratories. It automatically plots diffusion current as a function of voltage. The data-taking is done by a Speedomax Microampere Recorder, which provides greater speed of analysis and more complete detail of test results. A typical test run can now be made in about half the previous time. A full range of variation in test technique is available through a set of controls, all centralized in one panel. Step-by-step instructions for operation, testing, and calibration are included on the front of the instrument.

**Interchemical Rotational Viscometer**—Precision Scientific Co., 3737 W. Cortland St., Chicago 47, Ill. This instrument has been completely redesigned with all operating parts within one housing. The housing table top contains controls and dials within easy reach of the operator, making the apparatus simpler to manipulate. The viscometer has a broad utility and will give complete data on the flow properties of greases, paints, printing inks, plastics, adhesives, food products, and many other similar industrial products.

**High Consistency Rotational Viscometer**—Precision Scientific Co. This was developed in cooperation with The Texas Company to measure the viscosity of asphalt and related viscous substances. It makes possible a complete evaluation of the high consistency materials having complex flow characteristics. When testing high consistency liquids, it will determine the viscosity, the degree of flow complexity, and the relative elasticity. The viscometer was designed primarily for testing roofing, paving, and waterproofing products but is also suitable for testing viscous paints, varnishes, adhesives, and plastics. It measures and records viscosity from 0.001 to 1000 megapoises at various rates of shear. The sample cup can be rotated at any one of ten speeds from 0.0696 to 2200 revolutions per hour and these speeds are in direct relation to the rate of shear. Speeds can be changed by shifting a lever on the front panel.



# Speed of Testing

## A Summary Prepared by F. G. Tatnall\* for A.S.T.M. Committee E-1, Section on Effect of Speed of Testing

EDITORS NOTE.—At the Thirteenth Session of the Fifty-first Annual Meeting of the Society held in Detroit, Mich., June 21 to 25, 1948, a Symposium on Speed of Testing was presented. In addition to the data covered in the formal papers in the symposium, the committee was aware that there was a considerable amount of data that would be a valuable addition to the symposium but which data would not lend itself too well to formal papers. Mr. F. G. Tatnall agreed to summarize these data. This information was not available for inclusion in the 1948 *Proceedings* with the papers of the symposium but is included in the reprint of the symposium available in separate pamphlet form. The summary is included in this issue to Supplement the *Proceedings*.

### INTRODUCTION

By P. G. McVetty, Westinghouse Research Laboratories, Chairman of Section on Effect of Speed of Testing of Committee E-1

In addition to the formal papers presented at the 1948 Symposium on Speed of Testing, there is available a considerable amount of data relating to speed effects in tests of a wide variety of materials. In most cases these data are not suitable for additional papers or discussions of the five papers presented. They do, however, represent valuable information which supplements the Symposium papers and broadens the field covered.

Mr. F. G. Tatnall, who has actively supported the work of the section since 1934, volunteered to collect and summarize these data. By study of the literature, and by direct contacts with and letters to members of the section and others, Mr. Tatnall has collected the following notes from the sources indicated.

This summary of the latest information on speed of testing is arranged in small space for easy assimilation so that "he who runs may read." Some of these data have not yet been formally published. The names of contributors are noted in parenthesis.

### FERROUS METALS AND ALLOYS

#### Carbon Steel (L. H. Fry):

In A.S.T.M. *Proc.* (1940) Moore and Jones show that 800 per cent rate change has no serious effect on tensile and yield strengths. In a report to Committee A-1, Subcommittee XIII, in 1940, Mr. L. H. Fry shows that a three-fold increase in speed increases the yield point from 45,000 to 48,000 psi., which is of the same order of magnitude as the scatter of yield point determinations in tests of several specimens of the same material. Speed limits should specify either limiting rates of crosshead speed *under load* or rates of load application in pounds per square inch per minute. No high degree of precision is required in speed specifications since considerable variation can be tolerated in tests of carbon steels.

#### Ferritic Stainless Steels (R. H. Heyer, Armco Steel Corp.):

Tensile strength and elongation are not appreciably affected by testing speeds in the range available in commercial testing machines. The yield point, if

present, can be changed by speed, inertia, and machine stiffness characteristics.

#### Note on Effect of Speed of Testing on the Tensile Properties of Austenitic Stainless Steels<sup>1</sup> (R. H. Heyer, Armco Steel Corp.):

The tensile properties of most of the austenitic stainless steels are sensitive to the rate of strain in the tension test. Low strain rates result in high values of both tensile strength and elongation. The yield strength is not appreciably affected.

Crosshead speed, either free running or under load, is not a good measure of rate of strain because of the marked tendency of these materials to stretch in the shoulder sections and well into the grips. Rate of strain in the gage marked section or possibly time of test between yield and fracture can be used to control speed of testing.

The effect of strain rate is related to the temperature rise in the specimen. Controlled temperature tests show that the strength and elongation decrease as the temperature increases from atmospheric to about 400 F. It is probable that tension tests at room temperatures at very low strain rates approach isothermal conditions which result in the development of abnormally high properties.

<sup>1</sup> See R. H. Heyer, "Effect of Speed of Testing on the Tensile Properties of Austenitic Stainless Steel Sheet," ASTM BULLETIN, No. 158, May, 1949, p. 57 (TP 83).

### Related Technical Papers:

See 1947 A.S.T.M. Partial Bibliography on Effect of Speed of Testing, and particularly the basic investigation by J. Hammond Smith on "Rate of Elongation in Tension Tests," A.S.T.M. *Proceedings*, Vol. 27, Part II, pp. 507-521 (1927).

1. Speed Control Characteristics of Various Types of Commercial Testing Machines.—Report to section by H. L. MacBride, National Forge and Ordnance Co., May, 1947. See also accompanying Table I.

2. Round Table Discussion on Speed of Testing.—Held at the Fiftieth Annual Meeting of the Society, 1947. (Published in mimeographed form).

3. "Methods and Equipment for Controlling Speed of Testing."—L. K. Hyde, O. S. Peters Co., A.S.T.M. *Proceedings*, Vol. 48, pp. 1129-1186 (1948).

4. "High Speed Tension Tests at Elevated Temperatures."—A. Nadai and M. J. Manjoine, Westinghouse Research Labs., A.S.M.E. *Journal of Applied Mechanics*, June, 1941. Discusses effects of range of deformation rates from 1 per cent in 10,000 sec. (2 hr. 47 min.) to 100 per cent in 0.001 sec. or a variation of one to one billion. Aluminum and copper at elevated temperatures show continuous increase in yield strength with strain rate. For ferrous metals and alloys, temperature differences alter the speed relation complexly.

### NON-FERROUS METALS AND ALLOYS

#### Aluminum Alloys (F. M. Howell, Aluminum Research Laboratories):

The values obtained for the mechanical properties of aluminum alloys are not seriously affected by variations in speed of testing, hence a considerable range in testing speed is permissible. Recommended rate of stressing up to yield strength—not more than 100,000 psi. per min. Above yield strength, the crosshead speed under load should not exceed 0.5 in. per in. of gage length per min. Satisfactory results can be obtained by limiting the rate of straining to not more

TABLE I.—TESTING MACHINES (TENSION AND/OR COMPRESSION) MEANS OF REGULATING TESTING SPEED.

DATA FURNISHED BY THE MANUFACTURERS, COMPILED BY MR. H. L. MACBRIDE, NATIONAL FORGE AND ORDNANCE CO., JUNE, 1949.

Make	Type (Hydraulic or Mechanical)	Speeds Available, in. per min. (except where otherwise indicated)		Means of Regulating Speed	Described in Maker's Catalogue
		For Tests	To Position Crossheads		
Amsler 20 000-lb. 40 000-lb. 60 000-lb. 100 000-lb. 200 000-lb.	Hydraulic	0 to 7 0 to 3½ 0 to 5½ 0 to 5 0 to 4	Depends on Capacity	Delivery Regulator	No. 4 No. 8 No. 14 No. 20 No. 30
Amthor		From 0.0 to 20			
Baldwin-Southwark		Stepless—varying from 2½ to 6 depending on capacity.			
		0 to 20			
		0 to 1½			
Detroit	Hydraulic	0 to 1½	5	Gear Pump—with pump discharge controls.	Bulletin UT-3 ST-1
	Universal				
Dillon	Model L—Universal—Mechanical	¼ to 10	ditto	Gear reduction, sprocket and chain, single speed.	L
	Model M—Universal—Mechanical	0 to 20	ditto	Graham variable speed drive.	M
Instron	Mechanical; Tension Compression	0.02 0.05, 0.10, 0.20, 0.50, 1.0, 2.0, 5.0, 10, 12, 20	20 and 2.0	Positional, servomechanism controlled by selsyns from synchronous motor. Chart speeds 0.2 to 50 in. per min., independently selected to secure range of extension magnifications. Load ranges: 2 g. to 1000 (or 5000) lb. full scale.	
National Forge	Mechanical—Compression (For Cartons, etc.)	¼ (Fixed—A.S.T.M. Std.)	6	Two-speed gear shift.	Brochure No. 491
Olsen	Mechanical	Infinite Variable 0.025 to 2	Variable up to 8	Electronic variable speed control.	
	Hydraulic	Infinite Variable 0 to 2	Approximately 10	Hydraulic pump, load valve and automatic regulator valve. Crosshead adjustment by motor drive.	Bulletin No. 30
Riehle	Mechanical Tension—Compression	0.015 to 20	Depends on capacity	Combination two-speed gear shift and control knob of Thymotrol unit.	
	Hydraulic—Compression	0.05 to 2.5	2.5	Multi-piston pump regulated by control valve.	
	Hydraulic Tension—Compression	0.05 to 2.5	12	Separate motor driven crosshead.	
NOTE.—Mechanical Machines up to 5000 lb. can be purchased with testing speeds up to 20 in. per min. For machines 10,000 lb. and above speed to testing up to 3 in. per min. and adjusting speeds up to 10 in. per min. are available.					
Scott	Mechanical for multi-fixed speeds	0.2, 0.4, 1, 2, 3, 6, 10, 12, 20	lb. per sec. or	Mechanical for multi-fixed speeds. Selsyn motor control.	CC Thymotrol
	Electrical for variable range	Various in a 20:1 ratio	grains per denier	Potentiometer end rpm. Tachometer.	
Testing Machines Inc.	Hinde and Dauch Crush Tester	¼ to 2 as specified by purchaser.			Circular
Thwing-Albert	Electro-Hydraulic Pendulum, minimum 100 g. maximum 1000 lb.	Stepless Pendulum—2 to 30		Calibrated valve opening.	Bulletin P-478
	Fixed Crosshead minimum 50 g. maximum 10,000 lb.	Fixed crosshead—0.02 to 30		Servo motor positioning valve, controlled by L 50 Sentronec recorder for constant rates of straining and stressing.	
Young	Hydraulic—Universal	0 to 8	1 to 2 ft. per min.	Rotary pump with suitable control. Crosshead adjustable by motor.	Bulletin 60-1 60-2
	Mechanical—Universal	0 to 20	1 to 2 ft. per min.	Variable voltage and Thymotrol.	Bulletin 30-2

than 0.01 in. per in. per min. up to the proportional limit. This rate gives a time of 24 sec. to reach a proportional limit of 40,000 psi. or 36 sec. to reach a yield strength of 40,000 psi. Beyond the yield strength the speed can be increased to give fracture in a few additional seconds.

#### Effect of Speed in Testing Zinc and Zinc Alloys (E. H. Kelton, New Jersey Zinc Co.):

Zinc and zinc alloys are deformed by relatively light static loads. These same zincs show unexpected resistance to much heavier dynamic loads. In extruding zinc the pressure required is greater than that necessary for metals of relatively greater tensile strength like brass. Zinc, therefore, is considered sensitive to speed of deformation.

The effect of testing speed on the tensile strength of the Zamak die-casting alloys is typical. In Zamak-3 (A.S.T.M. Alloy XXIII), tensile strength of as cast specimens varies from 41,900 psi. at 0.05 in. per min. head speed to 43,100 psi. at 0.30 in. per min.

Subcommittee IV of A.S.T.M. Committee B-6 recommended that the following statement appear with the Standard Specifications for Zinc-Base Die Castings (B 86-48) (see Appendix to the specifications):<sup>2</sup> "In the tension testing of die-cast zinc alloy specimens the rate of straining shall not exceed 0.25 in. per inch per min. measured on the gage length of the specimen."

#### Copper and Copper Alloys (G. R. Gohn, Bell Telephone Labs., Inc.):

For sand castings, continuous castings, strip and rod, testing speeds up to a rate of straining of 0.50 in. per in. per min. have no significant effect upon tensile strength or elongation. At rates of straining as low as 0.025 in. per in. per min., difficulty is experienced in accurately recording data for yield strengths corresponding to 0.5 per cent elongation under load, as well as for 0.2 per cent offset yield strength.

#### The Effect of Speed on the Tensile Properties of Lead (A. A. Smith Jr., American Smelting & Refining Co.):

Because soft lead and many lead alloys spontaneously recrystallize at room temperature, there is little, if any, work hardening effect. Consequently, the speed of testing is very important and affects the absolute values very materially. As shown by the accompanying Fig. 1, the tensile properties decrease rapidly

<sup>2</sup> 1948 Supplement to Book of A.S.T.M. Standards, Part I-B.



with a decrease in testing speed. Also, the elongation tends to decrease, but at a less rapid rate. These data apply directly to soft lead only, but they apply to all lead alloys to some degree. As the rate of testing approaches zero, the tensile properties are very low, being of the order of 400 or 500 psi. if two or three years are taken for the testing time.

## CEMENT AND CONCRETE

**Portland Cement** (J. R. Dwyer, National Bureau of Standards):

The loading up to one half of the expected maximum load on the specimen may be at any convenient rate, after which the machine shall be adjusted to load at a rate in pounds per minute which shall be not more than  $1\frac{1}{2}$  times, and not less than one half of the expected maximum load on the specimen in pounds; thereafter, no change shall be made in the setting of the rate of loading controls until after the maximum load has definitely been reached, and the load is to be released. This applies to compression testing. An indication of the lack of sensitivity of cement and concrete to loading rate is shown by the present test method for compression testing of cement cubes which calls for a rate of loading not less than 1000 nor more than 6000 psi. per min.

For tension testing of the cement briquet, the rate of loading shall be  $600 \pm 25$  lb. per min.

For flexure testing of cement and concrete beams the loading rate shall be not greater than 150 psi. per min.

**Effect of Speed of Testing on the Compressive Strength of Concrete** (F. E. Richart, University of Illinois):

Relatively few studies have been made on the effect of testing speed on the strength of concrete. Since this material is subject to creep at very low loads, the effect of rate of loading might be expected to differ markedly from that for materials with a high proportional limit.

Reasonably consistent results were obtained in tests by Jones and Richart in 1936,<sup>3</sup> on the relation between speed of testing and the compressive strength of concrete. Tests were made by loading 6 by 12-in. cylinders to failure in periods of 1 sec., 5 sec., 20 sec., 1 min., 2 min., 10 min., 30 min., 1 hr., and 4 hr., thus producing a range in stress application varying from 3870 down to 0.12 psi. per sec. Both load and strain were measured by means of telemeter gages of the

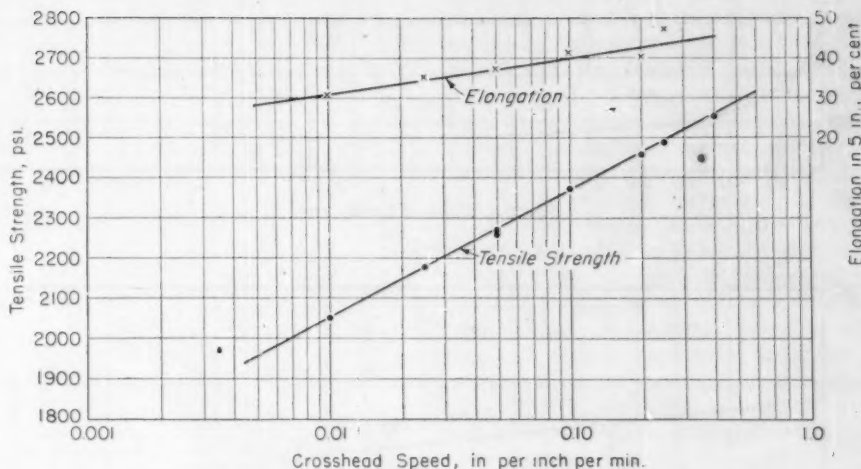


FIG. 1.—Effect of Speed on Tensile Strength and Elongation of Soft Lead at Room Temperature.

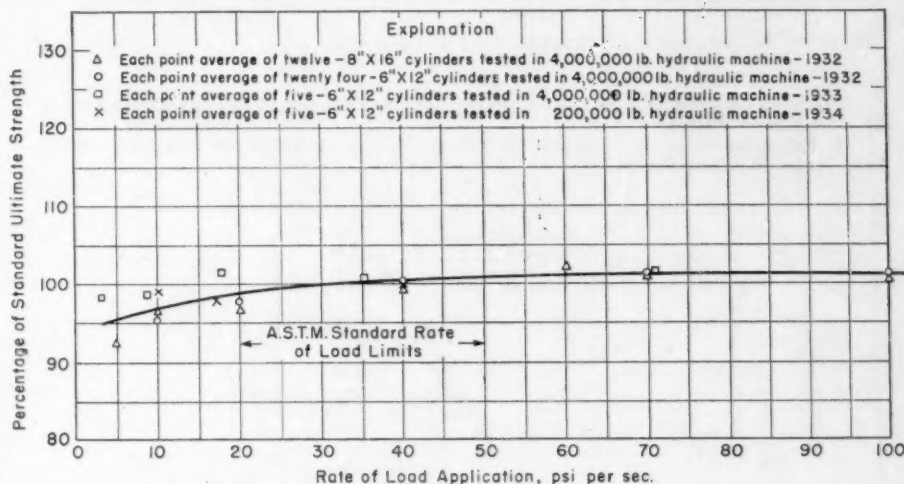


FIG. 2.—Ultimate Strength of Concrete is Reduced By Slow Rate of Load Application.

carbon resistor type. For all but the very slow loading rates, autographic records of load, strain and time were taken by means of an oscillograph, equipped with super-sensitive elements. Tests were made on three grades of concrete, at ages of 7 and 28 days.

The test results indicate that the strength increased with increase in rate of loading. The ratio of strengths obtained with the highest and lowest testing speeds was about  $1\frac{1}{2}$  to 1.

When the compressive strength was plotted against the loading rate, using a logarithmic scale for the latter, the test values were represented fairly well by straight lines. The straight lines correspond to the equation:

$$S = S_1 (1 + k \log_{10} R)$$

wherein

$S$  = strength at a given rate of loading,  
 $R$  = rate of applying stress in pounds per square inch per second,

$S_1$  = strength at the rate of applying stress of 1 psi. per sec., and

$k$  = a constant; about 0.07 for the 7-day tests and 0.08 for the 28-day tests.

It was noted in the paper that a similar logarithmic relation had been observed between modulus of rupture and rate of loading in tests of plain concrete made at the University of Wisconsin in 1933. A few other scattering tests agreed qualitatively with the conclusion from the above tests, though specific comparisons could not be made.

Similar data from the Bureau of Reclamation showing the effect of rate and duration of loading are given in Figs. 2 and 3.

## PLASTICS

**Effect of Speed of Testing on Molded and Laminated Materials** (A. G. H. Dietz, Massachusetts Institute of Technology):

<sup>3</sup>P. G. Jones and F. E. Richart, "The Effect of Testing Speed on Strength and Elastic Properties of Concrete," *Proceedings, Am. Soc. Testing Mats.*, Vol. 36, Part II, pp. 380-392 (1936).

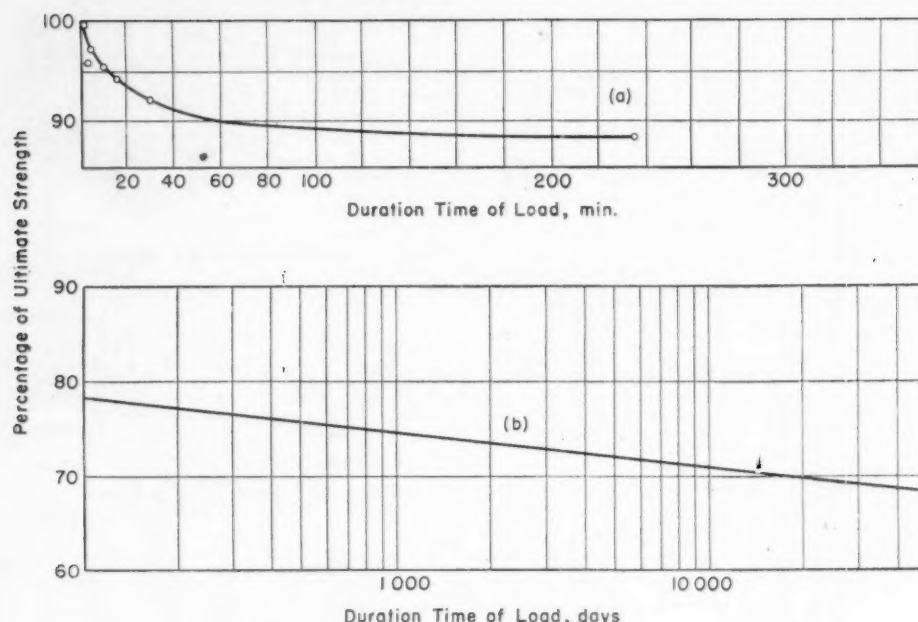


FIG. 3.

(a) Shows Concrete Will Support Ultimate Load as Determined by Standard Procedure for Only a Short Period.  
(b) Extrapolation of (a)—Concrete Will Support Only About 70 per cent of Ultimate Load for an Indefinite Period.

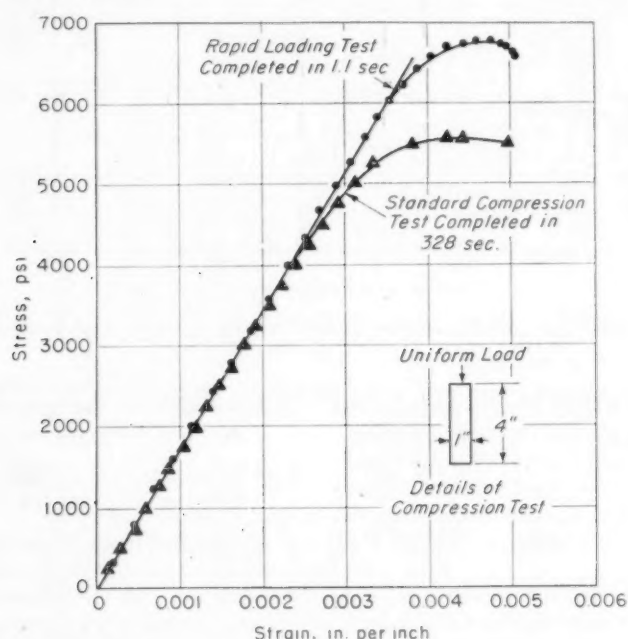


FIG. 4.—Stress-Strain Curves from Two Matched Douglas-fir Specimens 1 by 1 by 4 in. Long. The Increase in Strength Due to Rapid Loading was 22 per cent of the Standard Compressive Stress. Moisture Content 12.8 per cent. Specific Gravity 0.43 (Based on Weight when Oven Dry and Volume at Test). Specimen 39-59-86.

**Tension.**—At constant rates of cross-head motion:

Cellulose acetate butyrate, upper yield point, ordinary tensile stress.

Type	Crosshead Speed, in. per min.	Yield Point, psi.
H <sub>a</sub> .....	0.02 to 2.56	5200 to 6600
MH.....	0.02 to 2.56	3500 to 4200
S.....	0.02 to 2.56	2100 to 2800

At constant rates of ordinary strain:

Cellulose acetate butyrate, upper yield point, true tensile stress.

Type	Strain Rate, in. per inch per min.	Yield Point, psi.
H <sub>a</sub> .....	0.042 to 0.400	6300 to 7700
MH.....	0.049 to 0.395	4500 to 5500
S.....	0.049 to 0.450	2800 to 3400

**Bending.**—At constant rates of load, maximum bending stress:

Material	Load Rate, lb. per min.	Stress, psi.
CAB H <sub>a</sub> .....	4 to 1500	9 000 to 12 000
MH.....	4 to 1500	7 700 to 9 000
S.....	4 to 1500	4 000 to 5 300

Directional phenolic paper laminate.....	4 to 1500	18 800 to 25 000
Phenolic fabric laminate.....	4 to 1500	16 200 to 20 000

**Compression.**—At constant rates of crosshead motion, maximum ordinary compressive stress:

Material	Crosshead Rate, in per min.	Stress, psi.
CAB H <sub>a</sub> .....	0.04 to 10	7 800 to 11 500
MH.....	0.04 to 10	5 500 to 7 500
S.....	0.04 to 25	3 000 to 4 500

Directional phenolic paper laminate.....	0.04 to 2.5	28 000 to 33 000
Phenolic fabric laminate.....	0.04 to 2.5	27 500 to 31 500

## WOOD

**Effect of Testing Speed on Strength of Wood** (L. J. Markwardt and J. A. Liska, U. S. Forest Products Laboratory):

Figure 4 shows stress-strain curves for two compression tests at testing speeds varying in the ratio of 300 to 1.

Figure 5 shows a semi-logarithmic plot of stress versus time to failure at a constant rate of deformation. (While there is considerable scatter, the trend of the curve indicates an exponential relation.)

Figures 6 and 7 show how characteristics of various types of testing machines modify the speed-of-testing factor. In the case of mechanical-drive machines the actual rate of loading in the early portion of the test is appreciably less than the free-head speed because of lost motion and strain in the loading system. As the test progresses and the proportional limit is passed, the rate of loading approaches the free-running crosshead speed. Hydraulic machines under heavy loads show a decrease in loading rate in the latter portion of the test. In some cases with hydraulic machines, control valve adjustments must be made during the test to prevent the machine from stalling.

TABLE II.—VARIATION OF BREAKING STRENGTH AND ELONGATION WITH TESTING SPEED—RAYON.

1. Constant Rate of Loading Tester (Incline Plane), Capacity  $\frac{1}{2}$  to 20 kg.:  
0.07 per cent increase in strength per each sec. decrease in testing time, 5' to 100 sec., conditioned tests (yarns and cords).  
0.12 per cent increase in strength per each sec. decrease in testing time 5 to 100 sec., wet tests (yarns).  
0.50 to 0.65 per cent increase in strength per each g. per denier<sup>a</sup> per min. increase

<sup>a</sup> Denier is defined as the weight in grams of 9000 meters of test material at the standard conditions of temperature and humidity.



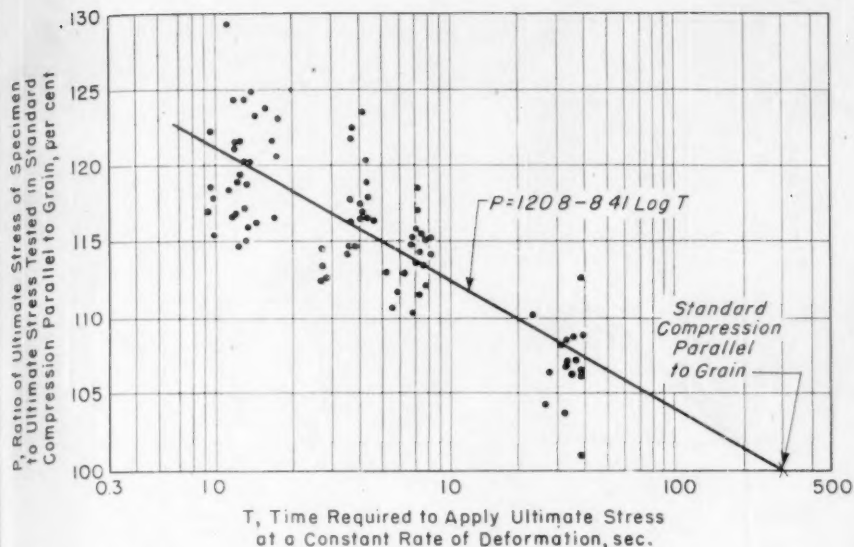


FIG. 5.—Effect of Rapid Loading on the Compressive Strength Parallel to the Grain of Sitka Spruce Specimens 1 by 1 by 4 in. Long. Moisture Content 12 per cent. Specific Gravity 0.32 to 0.42 (Based on Weight when Oven Dry and Volume at Test). Each Point Represents an Individual Test.

in loading rate (2 to 18 g. per denier per min.), conditioned tests (yarns and cords).

0.27 per cent increase in strength per each g. per denier per min. increase in loading rate (2 to 18 g. per denier per min.) wet tests (yarns).

0.37 per cent (relative) increase in elongation at break per each g. per denier per min. increase in loading rate (2 to 18 g. per denier per min.) conditioned tests (cords).

## 2. Constant Rate of Extension Tester—Schaevitz Differential Transformer—Cantilever Weighing Mechanism:

0.13 per cent increase in strength per each sec. decrease in testing time in the range 5 to 100 sec., conditioned tests (cords).

0.75 per cent increase in strength per each in. per min. increase in extension rate in the range 2 to 12 in. per min. for conditioned tests on a 10-in. specimen length (cords).

No positive increase in elongation at break in the extension rate range of 2 to 12 in. per min. (cords).

The above values are approximations only since the relationship between strength and elongation with respect to testing rate is not linear.

It has been customary to specify speed of testing of wood specimens in hydraulic machines as rate of crosshead motion under load in inches per minute, with a maximum allowable variation from specification speed of plus or minus 25 per cent. In mechanical machines, no-load speed of crosshead is used instead of speed under load.

## TEXTILES

Effect of Speed of Testing on Rayon and Cotton Yarns, Cords, and Fibers (N. M. Masich, E. I. du Pont de Nemours and Co.):

Tensile data secured on constant rate of loading or constant rate of extension

type strength testers indicate that the breaking strengths of rayon yarns and cords increase with an increase in the rate at which the load is applied during a test. The strength varies linearly with the logarithm of the rate of load application or rate of extension of the test specimen, whichever type of testing machine is used. This strength increase is of the order of 25 to 50 per cent when the testing time is reduced from 14 sec. to approximately 0.001 sec. and depends on the material being tested. For a constant rate of loading type tester elongation at break of viscose rayon cords under certain conditions of testing also increases logarithmically with the rate of load application while the elongation at the 10-lb. load level decreases logarithmically with loading rate. Table II, attached, lists the approximate variations of breaking strength and elongation with testing speed for various types of strength testers.

Tests on the strengths of cotton tire

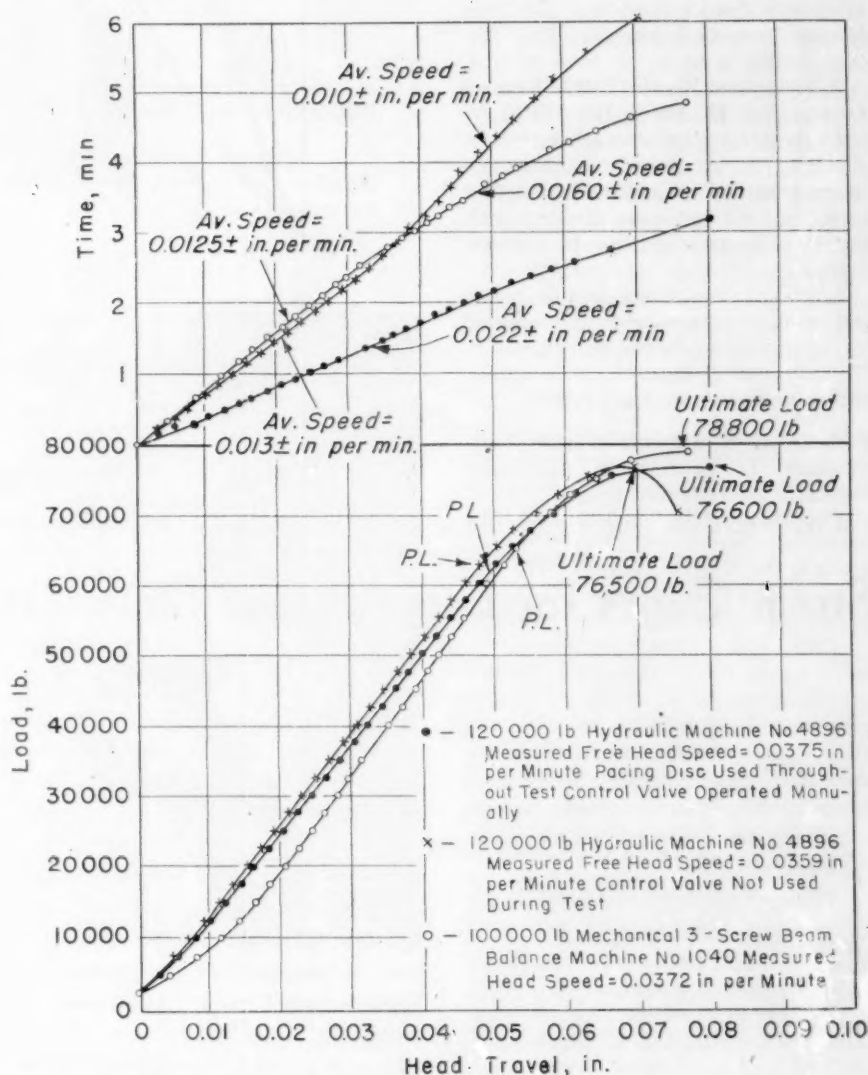


FIG. 6.—Head Travel - Load and Head Travel - Time Curves for 1 by 1 by 4 in. Douglas Fir Compression Parallel Specimens.

cords as run on an incline plane type tester by M. Castricum and A. N. Benson also show that the logarithmic relation between strength and rate of testing exists (A.S.T.M. *Proceedings*, Vol. 41, p. 1214 (1941)).

The effect of testing speed on the apparent strength of cotton fibers and yarns has been investigated by Midgley and Peirce (*Journal of the Textile Institute*, Vol. 17, p. T 330 (1926)). These authors find that a strength increase of 20 to 30 per cent is secured when the test yarn is broken in  $\frac{1}{4}$  sec. on a pendulum type tester over that observed at the standard test conditions requiring 20 sec. to break. An increase of approximately 43 per cent is secured when the testing time is reduced to  $\frac{1}{50}$  sec. The strength was also found to vary linearly with the logarithm of the testing time over a very broad time scale. No change in elongation at break is reported.

The effect of testing speed on the apparent strength of cotton and staple rayon fibers as tested on a Pressley Machine is also discussed by J. K. Phillip (*Textile Research Journal*, Vol. 19, p. 274, May, 1949).

Hindman and Krook (*Textile Research Journal*, Vol. 15, No. 7, July, 1945) describe a strain gage strength tester for obtaining impact-test data. These authors report that the apparent strength is increased for a high speed tensile test although elongation at break is definitely decreased.

Additional impact test data on cotton and nylon parachute webbing is reported by Stang, Greenspan, and Newman (*R.P. 1710, Journal of Research*, Nat. Bureau of Standards, Vol. 36, April, 1946).

Professor E. R. Schwarz (Textile Technology, Textile Division, Massachusetts Institute of Technology):

Reports that low, intermediate and

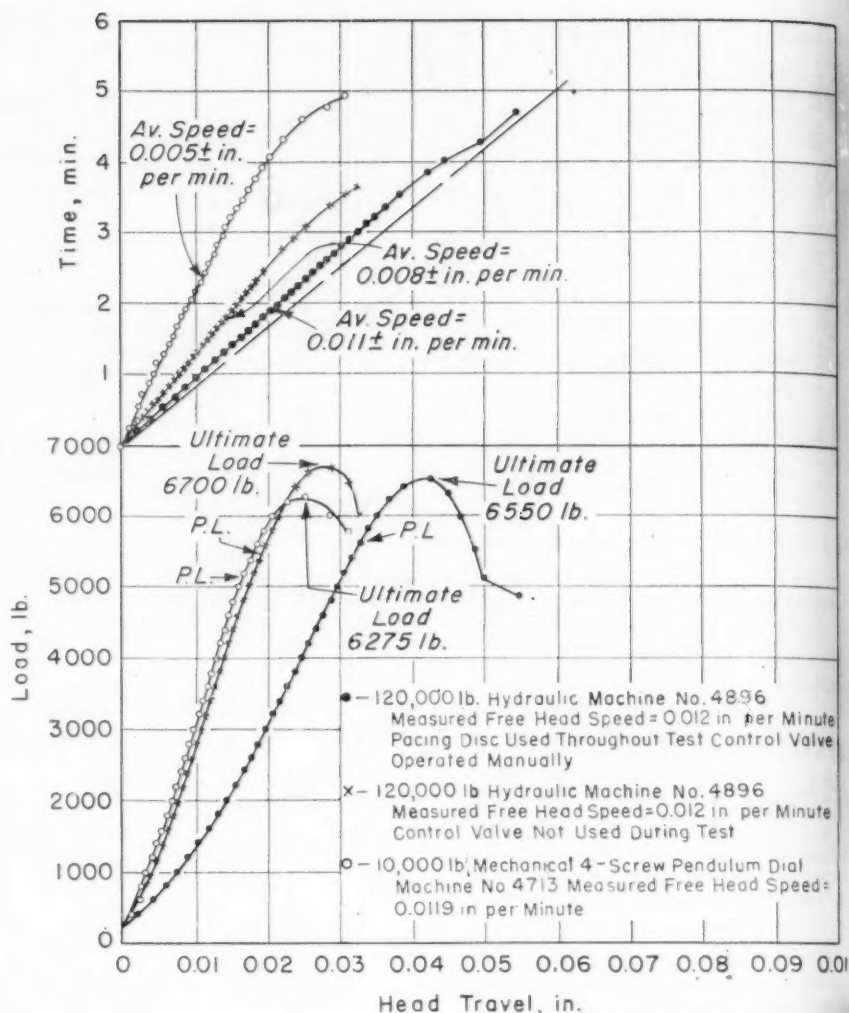


FIG. 7.—Head Travel - Load and Head Travel - Time Curves for 3 by 4 by 12 in. Douglas Fir Compression Parallel Specimens.

high-speed (impact) tests are made regularly at Slater Laboratories. A considerable amount of information has been col-

lected for the Army Air Corps as well as data resulting from an M.I.T. testing program.

## Strain Gages for Large Plastic Strains and Combined Stresses

By Joseph Marin<sup>1</sup>

IN RECENT years, several research investigations have been undertaken to determine the plastic stress-strain relations for materials subjected to combined stresses.

This paper describes two specially designed strain gages used for measuring

plastic strains on tubular specimens subjected to combined stresses. One of these strain gages (a tension-torsion gage) is a mechanical gage designed to measure angles of twist and axial strains on a tubular specimen subjected to axial tension and twisting. The other strain gage (a tension-tension gage) is a clip type electrical strain gage used for measuring longitudinal and lateral strains in a tubular specimen subjected to combined internal pressure and axial load.

### TENSION-TORSION STRAIN GAGE

Figure 1 is a photograph of the tension-torsion strain gage of which the details of construction are shown in Fig. 2. The gage consists of two rings,  $R_1$  and  $R_2$ , which are attached to the tubular specimen,  $S$ , by means of three rods spaced 120 deg. apart,  $P$ . The rods have conical ends and are connected to the rings with preloaded helical springs,  $H$ , so that compression in the springs is maintained throughout a test. In this manner, the rings do not slip relative to the specimen during the reduction in specimen cross-section in the plastic

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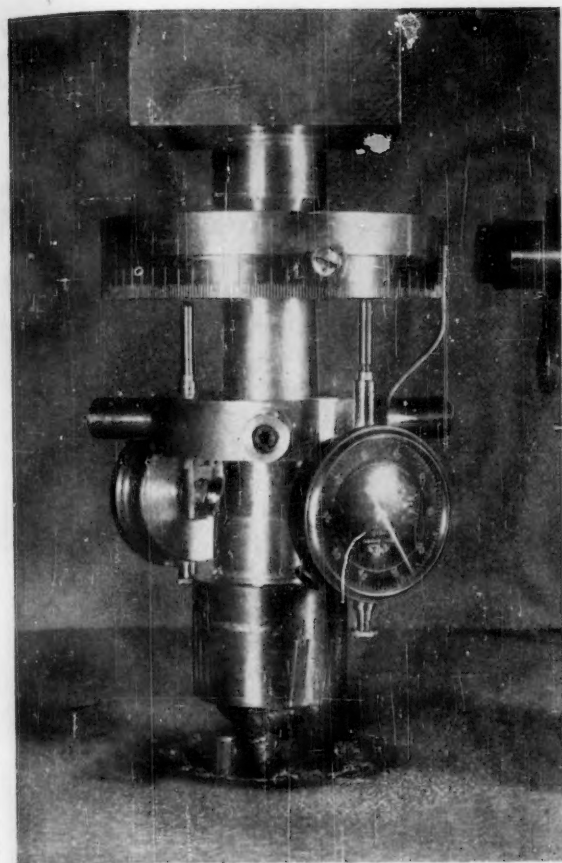


Fig. 1.—Tension-Torsion Strain Gage.

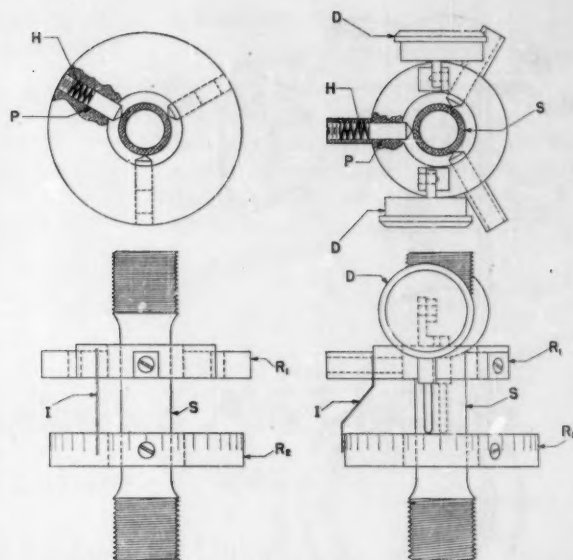


Fig. 2.—Mechanical Strain Gage for Measuring Large Plastic Angles of Twist and Axial Strains.

An indicator, *I*, attached to the top ring is used to measure the angle of twist on the lower ring. The measurement of the angle of twist is made possible since the lower ring is scribed with degree division marks.

The main features of the tension-torsion strain gage described in the foregoing are that it can measure both angles of twist and axial strains and that it is designed so that large strains can be measured at stresses beyond the ultimate stress of most materials.

#### TENSION-TENSION STRAIN GAGES

Figure 3 shows a photograph of a tubular specimen, *S*, with strain gages attached. These gages are used to measure large plastic strains in tubular specimens subjected to internal pressure and axial tension. Two kinds of gages are shown, one is used to measure axial strains and the other measures lateral or circumferential strains. The details of construction of both the longitudinal and lateral clip gages are represented in Fig. 4. The clip gages and rings were made of phosphor bronze strips  $\frac{1}{2}$  by 0.05 in. in cross-section. The longitudinal clip gage, *G*<sub>1</sub>, consists of a frame which is placed between two rings, *H*<sub>1</sub>, attached to the specimen. The rings are maintained in contact with the specimen by pointed rods and by the initial "spring" in the rings. Axial tensile strains are measured by the clip gage, *G*<sub>1</sub>, by means of electric SR-4 gages attached to the bridge of the clip (Fig. 5). Relatively large movements at the base of the clip produced by axial tension in the specimen, *S*, result in relatively small strains on the SR-4 gages at the bridge of the clip. The

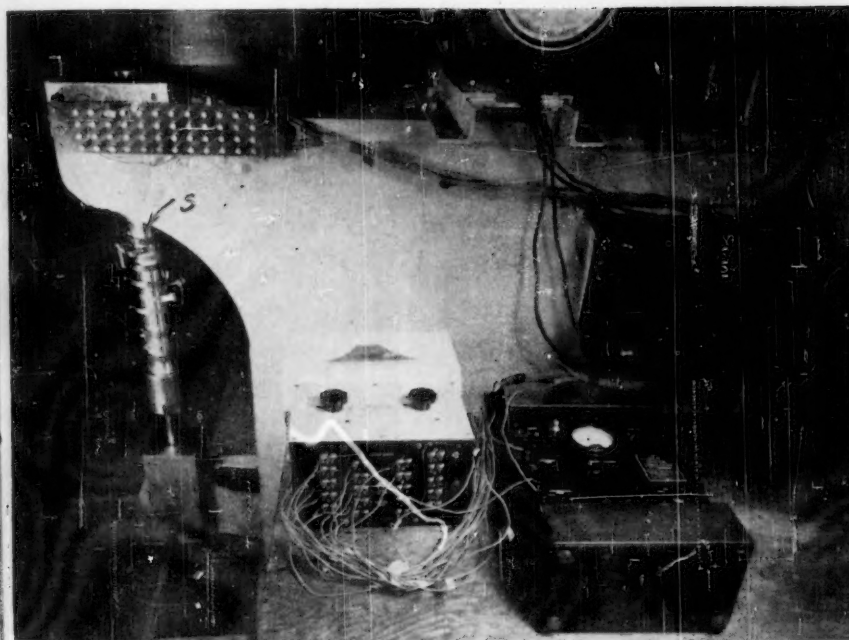


Fig. 3.—Tubular Specimen with Longitudinal and Lateral Clip Gages.

range. Dial gages, *D*, are attached to the upper ring, *R*<sub>1</sub>, and the plungers of the gages are in contact with the lower ring, *R*<sub>2</sub>. It is possible in this way to

measure the axial strains directly. Depending upon the magnitude of the strains to be measured, dial gages with  $\frac{1}{10,000}$  in. or  $\frac{1}{1000}$  divisions can be used.

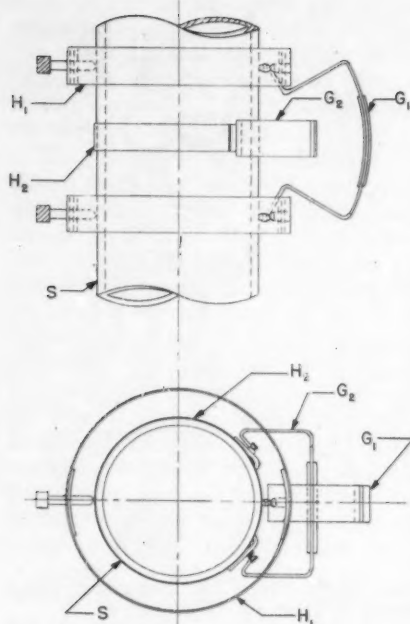


Fig. 4.—Electrical Clip Gage for Measuring Large Plastic Biaxial Strains.

strains produced in the SR-4 gages are measured by a strain indicator (Fig. 3). By cementing SR-4 gages on the upper and lower surface of the clip gage bridge, a temperature compensating gage is not

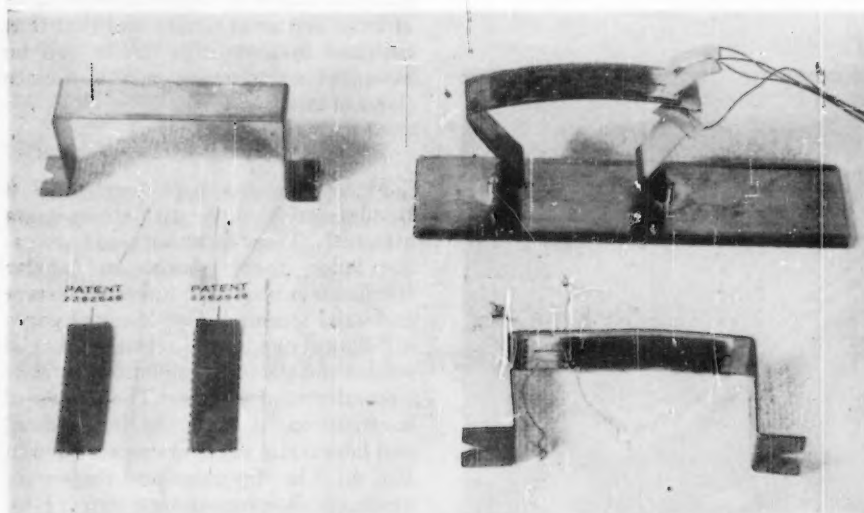


Fig. 5.—Longitudinal Clip Gage Showing Attachment of SR-4 Gages.

required, and increased sensitivity is obtained.

For the circumferential clip gage,  $G_2$ , the clip gage was attached to a thin metal band,  $H_2$ , under initial tension so that an increase or decrease in the specimen circumference could be measured by the SR-4 gages attached to the bridge of the clip gage.

The longitudinal clip gages were calibrated by using a vernier scale as illustrated in Fig. 6. By recording both the

actual movements at the base of the clip gage, as given by the vernier and as recorded by the SR-4 gages, the longitudinal clip gage could be calibrated. The lateral clip gages were calibrated by the stepped-tube device shown in Fig. 7. The stepped tube consists of an accurately machined tube with lengths of various diameters. By recording the readings of the SR-4 indicator for corresponding accurately known diameters of the tube, the calibration of the lateral

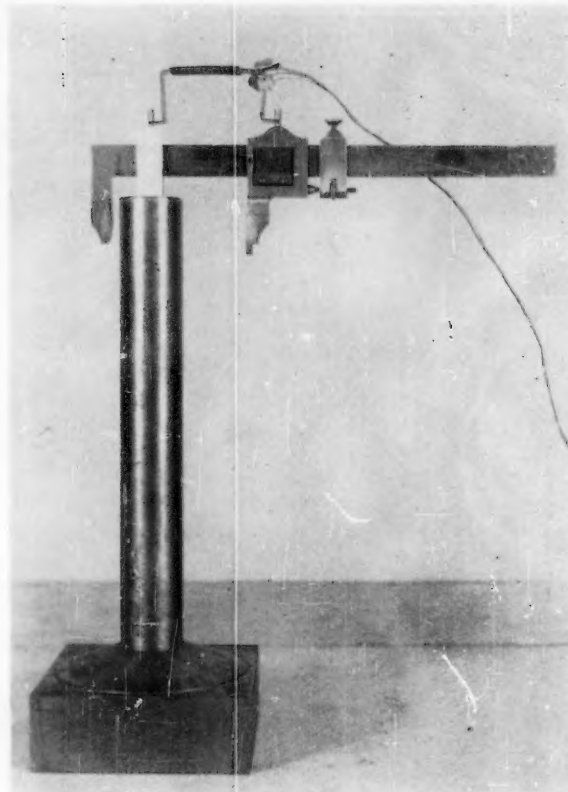


Fig. 6.—Vernier Used for Calibrating Longitudinal Clip Gages.

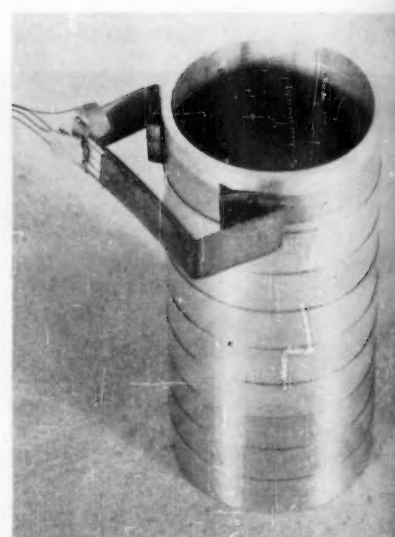


Fig. 7.—Stepped Tube Device Used to Calibrate Lateral Clip Gages.

strain gages was made possible. In an actual combined stress test, both the longitudinal and lateral-gages were applied with an initial pre-strain. The amount of pre-strain used depended upon the expected amount of reduction in specimen cross-section or increase in specimen length.

One of the main features in the design of the clip gages is the provisions that were made for attachment to the speci-

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men without using lugs or other devices. By attaching the gages to rings, the surfaces of the specimens were not influenced nor the properties of the material changed. That is, the welding or soldering of lugs by attachment of the clip gages would have affected the properties of the specimen.

The application of the foregoing strain

gages to combined stress tests is described in earlier papers by the author.<sup>2,3</sup>

<sup>2</sup> Joseph Marin, J. H. Faupel, V. L. Dutton, and M. W. Brossman, "Biaxial Plastic Stress-Strain Relations for 24S-T Aluminum Alloy," *Technical Note 1636*, Nat. Advisory Committee for Aeronautics, May, 1948, 96 pages.

<sup>3</sup> Joseph Marin, J. H. Faupel, and V. L. Dutton, "Tension-Compression Biaxial Plastic Stress-Strain Relations for Alcoa 24S-T," *Air Force Tech. Report 5694*, Army Air Forces, Air Materiel Command, April 30, 1948, 67 pages.

#### Acknowledgments:

The specially designed strain gages described in this paper were developed and used for projects which were sponsored and financed by the National Advisory Committee for Aeronautics and the United States Army Air Forces, Air Materiel Command.

## Some Observations on the Accelerated A.S.T.M. Life Test for Electrical Heating Wires

By Anton deS. Brasunas<sup>1</sup> and Herbert H. Uhlig<sup>1</sup>

**EDITOR'S NOTE.**—The original draft of this paper was presented at a meeting of Committee B-4 on Electrical Heating, Resistance, and Related Alloys held in New York, N. Y., June 8, 1949. This paper has been revised in accordance with discussion which developed at the meeting and in this form is being published in the ASTM BULLETIN.

IN CURRENT research on the fundamental mechanism of oxidation, we are using the A.S.T.M. Standard Method of Accelerated Life Test for Electrical Heating Wires (B 76-39)<sup>2</sup> for evaluating the oxidation resistance of several alloys. In choosing the test for this purpose, several advantages were recognized and the design and specifications scrutinized for possible improvement. As a result, one major and several minor changes in design were made. We also observed that humidity of the test atmosphere is an important factor, having actually greater significance in the test than some other factors now specified as requiring control.

#### Changes in Design of Test Chamber:

The major change we have made in the design shown in A.S.T.M. Method B 76-39 eliminates the mercury cup forming electrical contact at the lower terminal of the wire. The high vapor pressure of mercury at the temperature of the test seems to us a serious drawback in its use. Even at room temperature, the vapor pressure is high (0.001 mm. Hg), and at test-chamber temperatures (80 C.) during a run, the pressure may be 0.09 mm. or higher.

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<sup>1</sup> Corrosion Laboratory, Massachusetts Institute of Technology, Cambridge, Mass.  
<sup>2</sup> 1946 Book of A.S.T.M. Standards, Part I-B, pp. 226-235.

Both contamination of the test chamber with mercury and the fact that mercury vapor is toxic, particularly to individuals sensitive to it, make it desirable that mercury be eliminated. In

recent bulletins on experience with mercury poisoning<sup>3,4</sup> to recognize the serious consequences of neglect in this respect.

Our design makes use of a silver foil ribbon  $2\frac{1}{2}$  by  $\frac{1}{2}$  by 0.002 in. thick. This is attached, as shown in Fig. 1, to a 10-g. weight (corrected for the slight additional weight of the foil) by a screw, and is also attached to the side wall of the chamber. Silver does not oxidize in air; hence, the 10-g.

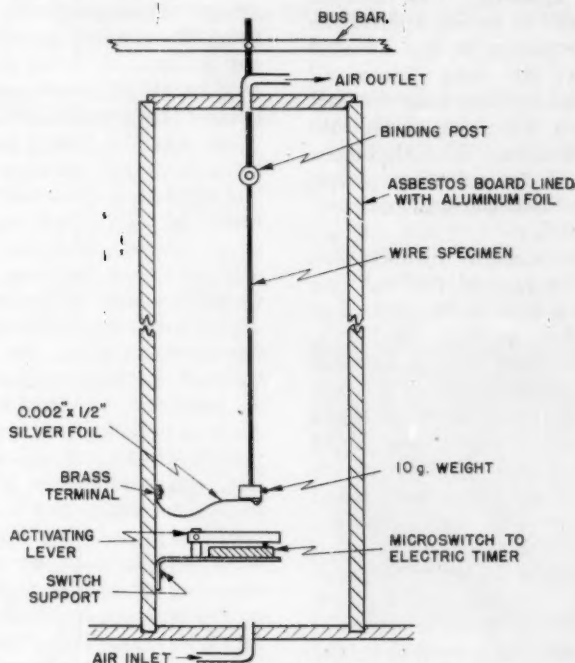


Fig. 1.—Variation of A.S.T.M. Apparatus for Life Testing of Electrical-Heating Alloys in Controlled Atmosphere. (Mercury Cup Replaced by Silver Foil.)

no way lessening the health hazard is the necessity for conducting the test in a partly closed room so as to avoid cross drafts, making adequate ventilation difficult. One has only to review

load on the wire does not change during

<sup>3</sup> Bureau of Mines Information Circular No. 7180.

<sup>4</sup> "Mercury," Ind. Data Sheet D-Chem. 17, National Safety Council, Inc., 20 North Wacker Drive, Chicago, Ill.

the test.<sup>5</sup> The foil, in turn, is sufficiently flexible to allow free extension and contraction of the wire during the heating and cooling cycles.

The test wire attached to the foil as shown can move in essentially two dimensions only, so that it is constrained to fall at time of failure onto a microswitch located a short distance below the weight. This, in turn, closes the circuit operating a signal light and simultaneously starts an electric clock indicating time of failure. We have also arranged that a relay is activated by the microswitch to throw in a dummy wire replacing the one burned out. This maintains a constant power load throughout the test and assures a more constant voltage across the remaining wires at all times.

#### Effect of Humidity:

It was necessary in our study to evaluate atmospheric humidity as one variable of oxidation resistance. Therefore, the quantitative effect of humidity in the A.S.T.M. test entered our program.

Control of the atmosphere surrounding the vertical wire specimen required sealing the test chamber to make it relatively airtight. The top and bottom of the chamber, accordingly, were closed and caulked, and adhesive tape was applied around all edges of the glass front. However, the transite sides were found to evolve appreciable moisture on exposure to the elevated temperature of the test; hence, all interior transite surfaces were covered with aluminum foil using a bakelite lacquer as adhesive. The aluminum foil, because of its reflecting power, also served to lower the wall temperature of the chamber.

Air of known constant humidity was supplied at the rate of 600 ml. per min.<sup>6</sup> through a tube at the bottom of the test chamber, as shown in Fig. 1, and allowed to escape through a similar tube at the top. This rate of air flow was sufficiently great to maintain an essentially constant partial pressure of oxygen at all times. Analyses of the air at the inlet and outlet were found to be 20.70 per cent and 20.55 per cent O<sub>2</sub>, respectively.

One advantage of the sealed chamber, in addition to permitting atmosphere control, is a more uniform temperature of the wires as compared to the

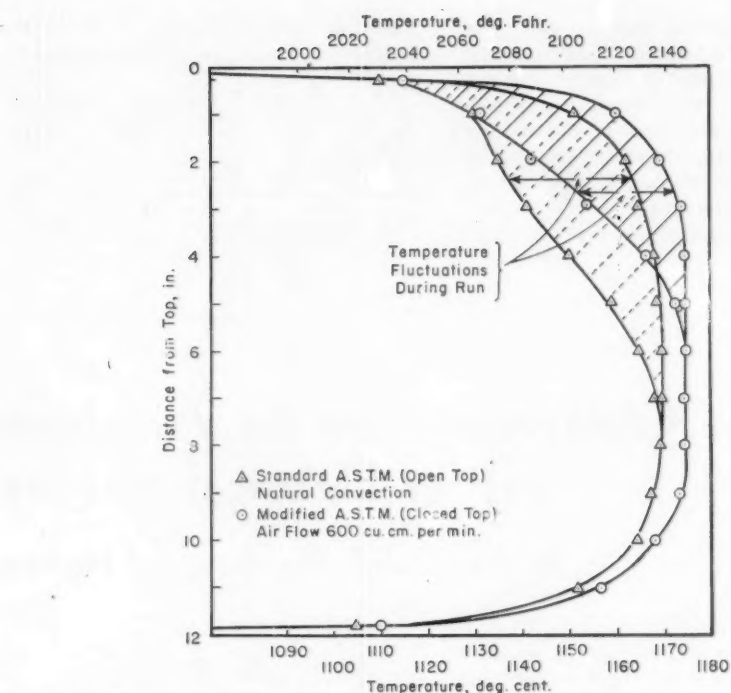


Fig. 2.—Variations in Temperature of No. 22-gage Wire (20 per cent Chromium, 80 per cent Nickel) at Constant Voltage as Observed in Open Top and Completely Enclosed Test Chamber. Fluctuations at Upper End Believed to Be Caused by Air Turbulence.

presently specified open-top chamber. The resulting improvement in temperature uniformity is illustrated in Fig. 2. To obtain "true" temperature from the apparent temperature as measured using the optical pyrometer, a total correction of 15 C. (27 F.) was used to account for emissivity and light transmission through the glass window.

Air was humidified by bubbling it through water contained in a flask immersed in an oil thermostat maintained at 25 C.  $\pm$  0.01 C. In other tests, air was dried by using suitable solid drying agents or by passing it through a trap cooled with acetone-dry ice mixture. Intermediate humidities were obtained by bubbling air through saturated salt solutions kept at constant temperature as above. Air so humidified or dried was checked periodically for actual humidity at the inlet and outlet of each test chamber using a calibrated hair hygrometer.

Although humidity has always been considered to play some part in the oxidation of metals at elevated temperatures, the quantitative effect in the A.S.T.M. Life Test turns out to be surprisingly high. It was found, for example, that at 1175 C. (2150 F.) a 20 per cent chromium, 80 per cent nickel wire from a commercial source had a useful life of 294 hr. in air saturated with water vapor, and a useful life of 518 hr. in dry air. This represents an increase in life of 76 per cent

owing to the lowered humidity of the atmosphere alone. Another wire of the same nominal composition, but from a different manufacturer, was tested, and an increase in life of 44 per cent resulted when similarly tested at 1175 C. (that is, 270 hr. versus 188 hr.). The actual causes for this difference of behavior require further data and experiments.

The important point is that humidity is an important factor in determining the behavior of wires in the A.S.T.M. Life Test. Frequently, a standard wire is run simultaneously with other wires to correct for changeable factors like humidity, but this is only partly corrective since the quantitative effect of humidity may be different for each wire, as noted above, even though the wires are of the same nominal analysis. Furthermore, variations in humidity during a given run, such as might apply to the open chamber, may produce, as we find in controlled tests, slightly shorter life than an average humidity without variation. With an effect so overshadowing, it seems to us that the only satisfactory arrangement is to control humidity in the test chamber within reasonable limits.

#### Acknowledgment:

This investigation was made possible through support of the Naval Bureau of Ordnance, Contract No. Nord 9661 to whom the authors express their appreciation.

<sup>5</sup> This 10-g. load on an 0.025-in. wire results in a stress of 45 psi. which increases gradually to approximately 120 psi. at failure because the cross-sectional area is progressively reduced by corrosion to approximately 0.015 in.

<sup>6</sup> 600 ml. per min. corresponds to an average theoretical atmosphere replacement every 20 min. for our unit.



# A Direct Reading Attachment for the Stormer Viscometer\*

By H. B. Klimper<sup>1</sup>, L. P. Larson<sup>1</sup>, and J. H. Calbeck<sup>1</sup>

THE device described in this paper provides for reading the consistency of paints directly on a calibrated scale in either "Grams Pull" or Krebs units and can be used with any Stormer viscometer equipped with a stroboscope. The purpose of this attachment is to speed up the task of running consistency measurements. It does not increase the accuracy nor sensitivity of a Stormer instrument; in fact, a slight but insignificant reduction in the accuracy or sensitivity of the device is the price paid for the greater speed.

The adaptation of the stroboscopic timer for use on a Stormer viscometer was outstanding and has greatly improved the speed and accuracy of Stormer consistency determinations.<sup>2</sup> Furthermore, its use in many laboratories has resulted in a shift from the usual Krebs units to reporting all determinations in "grams pull" according to A.S.T.M. Method D 562-47<sup>3</sup>. With the attachment described in this paper all readings are made at a spindle speed of 200 rpm, but instead of adjusting the weights on the usual weight holder to obtain the necessary "grams pull," this device provides a simple lever which is adjusted to obtain the end point, and then the results are read directly on a computing scale in either "grams pull" or Krebs units as may be required.

The principle of this device is that of the inclined plane apparatus found in every physics laboratory. Instead of the ordinary inclined plane a highly polished chromium-plated stainless steel rod  $\frac{1}{2}$  in. in diameter and 30 in. long is employed. Sliding on this rod is a weighted ball bearing sleeve which is attached to the cord of the Stormer viscometer and the necessary "grams pull" is quickly provided by adjusting the angle of inclination of the rod.

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\* Presented at a meeting of A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products held in Atlantic City, N. J., June 29, 1949.

<sup>1</sup> Research Laboratories, American Zinc Sales Co., Columbus, Ohio.

<sup>2</sup> This device known as the National Lead Co. Timer was invented by Peterson and Prane in 1946. See paper by E. P. Peterson and J. W. Prane, "Stroboscope Timer for Stormer Viscometer," Symposium on Paint and Paint Materials, Philadelphia Spring Meeting, Am. Soc. Testing Mats., p. 93 (1947). (Issued as separate publication, STP No. 75.)

<sup>3</sup> Standard Method of Test for Consistency of Exterior House Paints and Enamel—Type Paints (D 562-47), 1947 Supplement to Book of A.S.T.M. Standards, Part II, p. 183.

The attachment with the Stormer instrument removed is illustrated in Fig. 1. The long slide rod may be seen extending downward and to the right. Early at-

engages the brass quadrant to rotate it 5 deg. The combination of the ratchet handle, the thumb screw, and the brass quadrant makes possible the adjustment

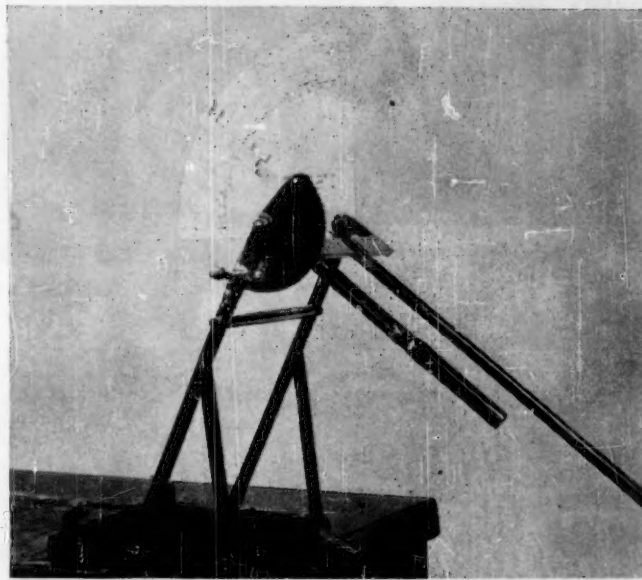


Fig. 1.—Attachment with The Stormer Instrument Removed.

tempts to make a frictionless slide weight were not successful, but late in 1947 a ball bearing sleeve was introduced by the Thompson Industries, Inc., of Manhasset, N. Y. The use of a  $\frac{1}{2}$  in. ball bearing sleeve on the slide rod made possible an almost frictionless slide weight. A phantom drawing of the ball bearing sleeve is shown in Fig. 2. The ball bushing is mounted in a brass cylinder to provide a total weight of 200 g., and this weighted sleeve is fitted to slide on the polished rod.

The apparatus consists simply of a heavy base which may be secured to a table by means of three screws. Attached to the base and extending upward and slightly to the right are two brackets 15 in. long. These brackets provide a support for a stationary brass quadrant and a large aluminum quadrant upon which the computing scale is printed. Between the two brackets and revolving about a horizontal axis is a brass yoke on a hinge which supports the slide rod and weight. Attached to the yoke is a ratchet lever which engages cogs on the brass quadrant whereby the angle of inclination may be adjusted. For a fine adjustment, a thumb screw

of the angle of inclination a matter of one or two seconds.

On the opposite end of the yoke is an aluminum bar which carries a fixed hair-line pointer and an adjustable sliding hair-line pointer. These pointers, together with the calibrated scale of the large quadrant, provide the direct read-

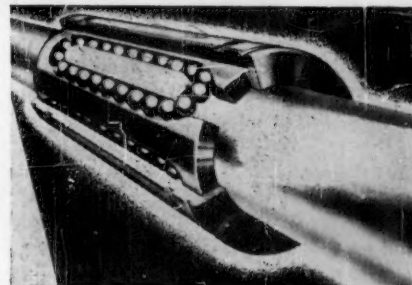


Fig. 2.—Phantom View of the Ball Bearing Sleeve.

ing feature of the attachment which will be fully described later.

To use the attachment a Stormer viscometer with stroboscope attached is slid into position on the heavy metal base and fastened tightly by two thumb-

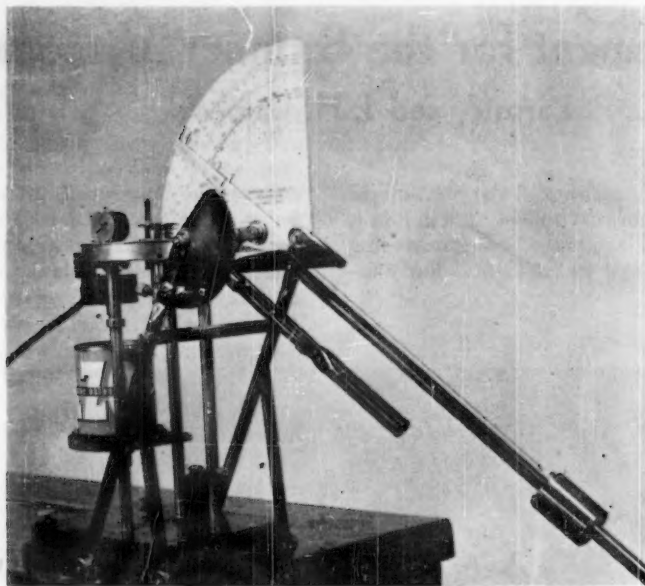


Fig. 3.—The Complete Apparatus Viewed from the Right.

screws. Figures 3 and 4 illustrate the apparatus with the Stormer instrument in place. The base should be properly leveled before use. The draw cord of the Stormer instrument is attached to an eye in the slide weight and pulled to the top position by cranking the winding drum with the brake "on." The stroboscope is connected to a light source, a submerged paddle attached to the spindle, and a can of paint adjusted to the proper level in the usual manner. The determination is made by simply releasing the brake and adjusting the angle of inclination with the ratchet lever and the thumbscrew until the end point is obtained on the stroboscope. This requires only a few seconds, and the slide weight will have traveled only a fraction of the distance down the inclined slide rod. If for any reason an end point is not obtained by the time the weight has traveled the length of the rod, a new start is made after cranking the weight to the top position again. The consistency of the paint is read on the computing scale in either "grams pull" or Krebs units.

It was found advisable to use a set of four different slide weights in order to provide conveniently a "grams pull" range from 0 to 1000 g. The accuracy and ease of operation of the instrument is greater when the lightest possible weight is used or, in other words, when the smallest angle of inclination is required. Although error due to friction increases as the slide rod approaches a horizontal position, this factor is not as important as the speed and ease of adjustment which is better when the angle of inclination is small. Therefore, a nest of weights was designed. The smallest

weight containing the ball bushing had a total weight of 200 g.; the second weight consisting of a cylinder of brass slides over the 200-g. weight to make a total of 400 g.; the third weight consisting of a larger brass cylinder slides over the 400-g. combination providing a total of 600 g.; a fourth weight for 800 g. and a fifth for 1000 g. Figure 5 shows three of the weights on the slide rod. In use, the three are nested and appear as one large cylinder. The nested weights can be quickly assembled or disassembled, each being held in position by a special spring clip.

The computing scale and pointers are shown in Fig. 6. There are six concentric scales on the quadrant. The scale on the largest diameter is one showing graduated degrees of arc. The fixed hair line on the pointer always indicates the angle of inclination in degrees. The next scale of shorter radius is used when a 1000-g. slide weight is necessary. This scale consists of a solid line with "grams pull" graduation on the outside and Krebs units on the inside of the line. Then in regular order come the scales for the 800-, 600-, 400-, and 200-g. weights. These scales are printed in color, and the sliding hair-line pointer should be adjusted to correspond to the weight being used. For example, the sliding hair-line pointer in Fig. 6 is on the 400-g. scale and should be used when the 400-g. slide weight is attached to the cord.

The assembled apparatus may be checked against a standard paint or against an oil by the method of Jackson and Madson.<sup>4</sup> If there should be any

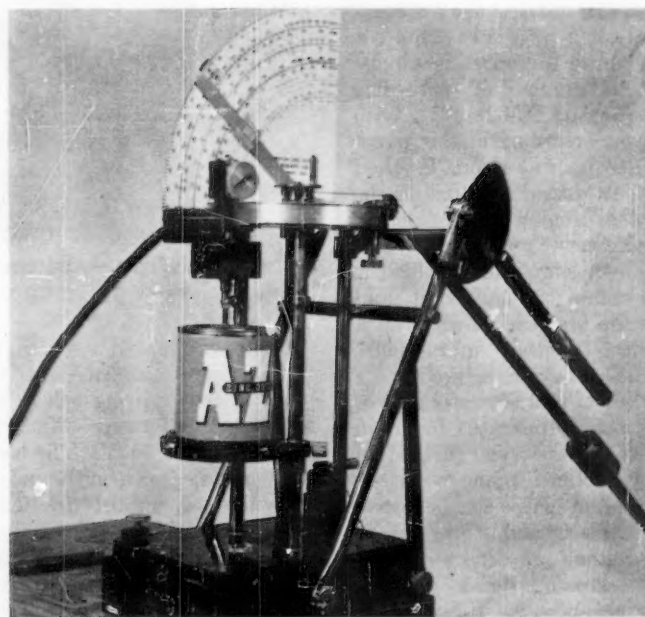


Fig. 4.—The Complete Apparatus Viewed from the Left.

doubt about the reliability of the slide weight, the method may be checked with a weight holder and slotted weights in the usual manner. Figure 7 shows the Stormer instrument with a weight holder and weights attached. The cord is removed from the slide weight, the slide rod is raised to an angle of about 45 deg., and the weight holder then swings freely when the cord is placed over the pulley. Either a stop watch or the stroboscope can be used in checking.

<sup>4</sup> C. F. Jackson and W. H. Madson, "A Method for Standardization of Krebs Modified Stormer Viscometers," see p. 36.



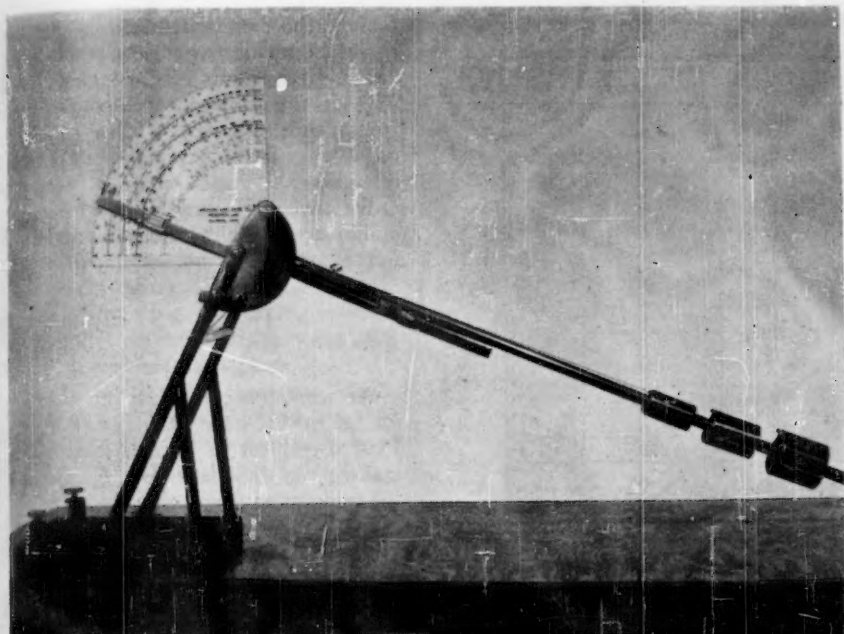


Fig. 5.—Slide Rod Showing Three Weights.

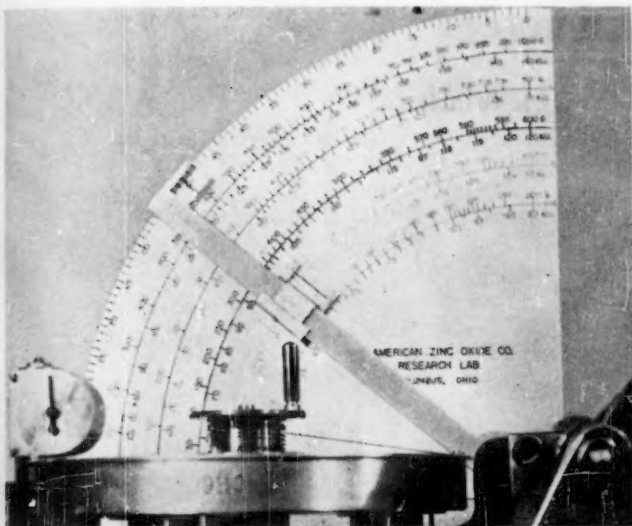


Fig. 6.—Computing Scale and Pointers.

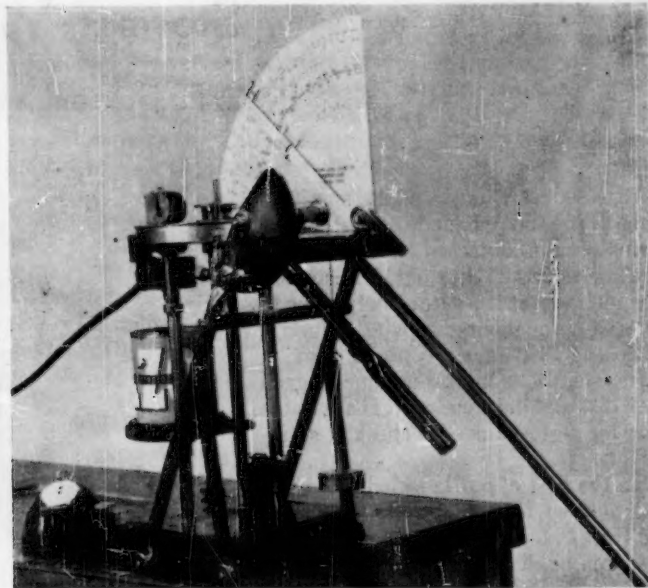


Fig. 7.—Complete Apparatus with Draw Cord Attached to Weight Holder for Checking.

TABLE II.—GRAMS PULL DEVIATION ± FROM EXACT CHECK.

Deviation	Frequency	Frequency × Deviation
15	1	15
9	1	9
8	0	0
7	0	0
6	2	12
5	5	25
4	4	16
3	11	33
2	44	88
1	32	32
0	136	0
-1	32	32
-2	28	56
-3	19	57
-4	4	16
-5	3	15
-6	2	12
-7	4	28
-8	2	16
-9	2	18
-10	2	12
-11	2	22
-12	1	12
-15	1	15
Total	338	541

$$\text{Average deviation} = \frac{541}{338} = \pm 1.60 \text{ g.}$$

TABLE I.—RANGE OF KREBS UNITS.

Range of Krebs Units	Frequency
135	1
130	2
125	1
120	3
115	2
110	3
105	5
100	19
95	16
90	58
85	72
80	53
75	62
70	28
65	3
60	2
55	6
Total	338

If a check indicates that the slide weight is running light, the bushing should be removed from the rod, washed with mineral spirits, dried and a drop of instrument oil put in each ball race and the bushing replaced. The rod should be kept free of oil and dust.

In order to determine the reliability and accuracy of this attachment, all the consistency determinations made in our laboratory for several months were made by first determining the consistency with the slide weight and then removing the slide weight and rechecking with the weight holder as shown in Fig. 7. In all,

338 duplicate determinations were made covering a range of Krebs units from 55 to 135 as indicated in Table I. For the heavier and lighter consistencies only a few determinations were made, but in the range between 70 and 100 Krebs units, inclusive, a total of 308 determinations were made and were fairly evenly distributed throughout this range. All determinations were made with a submerged paddle at 200 rpm. and at 85 F.

From the standpoint of "grams pull," Table II shows the frequency and deviation.

tion from an exact check of all of the 338 determinations. Although a number of the poor checks were repeated, all readings were included in this table and show an average deviation of only  $\pm 1.6$  g. Of the 338 determinations 136 check the vertical pull exactly. Sixty-four tests failed to check the vertical pull by 1 g. and 72 tests were 2 g. in error. Beyond this range, the errors were scattered but the over-all or average deviation of  $\pm 1.6$  g. indicates that this apparatus may be relied upon for exact research work.

Table III shows the deviation from the mean based on Krebs units. In this 281 determinations check the vertical exactly. The distribution pattern, however, is quite narrow and this is accounted

TABLE III.—KREBS UNITS DEVIATION  $\pm$  FROM EXACT CHECK

Deviation	Frequency	Frequency $\times$ Deviation
10	0	0
9	0	0
8	0	0
7	0	0
6	0	0
5	0	0
4	0	0
3	0	0
2	1	2
1	20	20
0	281	0
-1	30	30
-2	6	12
-3	0	0
-4	0	0
-5	0	0
-6	0	0
-7	0	0
-8	0	0
-9	0	0
-10	0	0
Total	338	64

$$\text{Average deviation} = \frac{64}{338} = \pm 0.19.$$

for by the fact that there is an interval of about five "grams pull" between Krebs units in this range. An average deviation of  $\pm 0.19$  shows that from the standpoint of this practical consistency unit the attachment is most reliable.

We have been using this device in our laboratory for eighteen months and have found it practical and convenient.

#### Acknowledgment:

We want to express our appreciation for the careful and patient work of Mr. Paul Dennison of our laboratory in making the duplicate tests from which the tables were prepared.

## A Method for the Standardization of Krebs Modified Stormer Viscometers\*

By C. F. Jackson<sup>1</sup> and W. H. Madson<sup>1</sup>

**M**ANY paint specifications in use today include consistency in terms of units obtained by the use of a Krebs Modified Stormer Viscometer. The instrument has accordingly come to be used throughout the paint industry. Since the instrument contains moving parts which can and do become clogged with dirt and dried paint, it is advisable that its performance be checked at intervals.

To the best of our knowledge, no formal method for the standardization of the Krebs Modified Stormer Viscometer has been published although some users have no doubt set up methods of their own, such as running a standard paint, for checking their instruments. This paper describes a method of standardization as well as the work done in its derivation.

Some data derived by J. A. Geddes and D. H. Dawson<sup>2</sup> indicated that the relationship between poises of hydrocar-

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\* Presented at a meeting of A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products held in Atlantic City, N. J., June 29, 1949.

<sup>1</sup> E. I. du Pont de Nemours and Co., Pigments Dept., Wilmington, Del.

<sup>2</sup> J. A. Geddes and D. H. Dawson, "Calculation of Viscosity from Stormer Viscometer Data," *Industrial and Engineering Chemistry*, Vol. 34, p. 163 (1942).



Fig. 1.—Equipment Used in This Study.



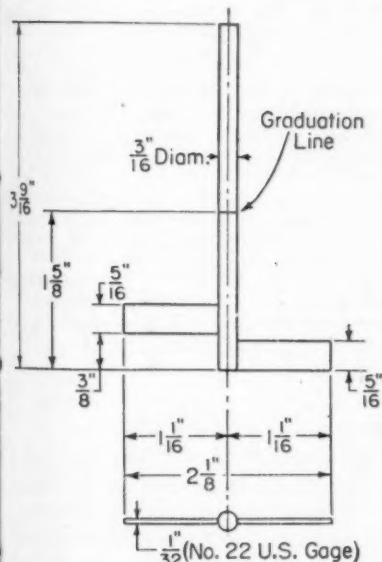
bon oils and grams per 200 rpm. on a Stormer instrument would be a straight line. If this indicated straight-line function were established, then a graph could be set up for use by the paint man in checking his instrument. It would be necessary for him to secure a sample of a hydrocarbon oil with known poise values at specific temperatures and run it on his Stormer instrument. Reference to the graph would tell him whether his instrument was in good working order.

A sample of standard viscosity oil "N" was secured from the National Bureau of Standards, Washington, D. C. The particular sample received had the following viscosity values as reported by the Bureau of Standards:

Temperature	Poises
20 C.	13.79
25 C.	9.34
37.8 C. (100 F.)	3.85

Measurements were made on the oil at each of the three different temperatures with several Stormer instruments from our laboratories. Since these instruments were equipped with stroboscopic timers, it was possible to secure readings to an accuracy of 1 g. The results obtained by plotting these data confirmed the indicated straight-line relationship of J. A. Geddes and D. H. Dawson.

At this point it must be made clear that, since such a line had to be set up with one instrument, any use of the line in checking a given Stormer instrument would mean that its performance was being compared with that of a "standard" instrument. To our knowledge,



All Dimensions Subject to a Tolerance of  $\pm 0.004$ " Material: Stainless Steel

Fig. 2.—Paddle-Type Rotor for Use with Stormer Viscometer.

there was no such "standard Stormer viscometer" in existence, and it became

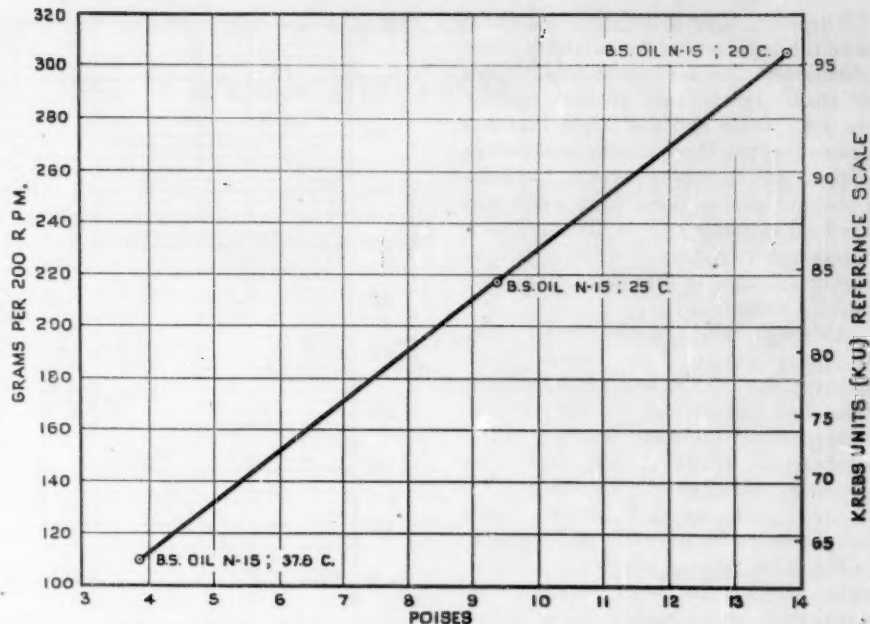


Fig. 3.—Grams per 200 rpm. versus Poises for B.S. Oil No. N-15.

necessary to establish one. It seemed most practical to do this by using the average results obtained from a group of new instruments which were known to be in excellent mechanical working order. This was done with the cooperation of Mr. Patterson, of the Arthur H. Thomas Co., who supplied five new instruments from stock and made available the facilities of his laboratory.

The temperature of the oil was controlled closely by means of the jacketed container shown in Fig. 1. This assembly consisted of a pint can for the oil which was enclosed on its sides and bottom with a sealed jacket (quart paint can) through which water of controlled temperature could be circulated continuously. With the jacket attached to a constant-temperature water bath with a circulating pump, the whole unit was operated as a closed system.

Throughout the entire study, a thermometer certified by the Bureau of Standards graduated to 0.2 C. and easily read to 0.1 C. was used. The results of a short study to determine the effect of temperature variations on the "Stormer viscosity" of the oil being used are given below:

Temperature, deg. Cent.	Stormer Units, g. per 200 rpm.	Krebs Units (K.U.)
24.0	237	87.3
24.5	230	86.3
25.0	224	85.4
25.5	217	84.4
26.0	211	83.5

These results show that, at least in the 25 C. range, 0.1 C. is equivalent to 1.3 g. at 200 rpm. with this sample of oil. They show that a 2 C. change in temperature is equivalent to practically 4 Krebs units on the oil.

The regulation submerged type of paddle, as shown in Fig. 2, was used in

the study. In order to observe the effect of variations in paddle dimensions on the accuracy of the determinations, the area of the blade faces of one paddle was reduced in steps by filing off at the blade ends. Stormer readings were made on the oil at constant temperature of 25 C. at each step with the following results:

Total Blade Length, in.	Stormer Units, g. per 200 rpm.
2.159	227
2.142	222
2.125	217
2.098	208

These data show that the paddles must be kept in good condition and that they must be checked, preferably with micrometer calipers, for accuracy of dimensions as a part of any standardization of the instrument on which they are used.

With all of these points checked and under control, readings were made on the sample of oil at the three different temperatures with the five new Stormer instruments mentioned previously. Whenever the temperature of the oil was changed, it was held at the new level for a minimum of 15 min. to insure complete temperature stability of the oil, before any Stormer readings were taken. Then the grams per 200 rpm. value was determined for each instrument, several check readings being taken on each to insure an accurate value. The data thus obtained are given below:

Oil temperature...	20 C.	25 C.	37.8 C. (100 F.)
Poises of oil used...	13.79	9.34	3.85
Viscometer			
Stormer Units, g. per 200 rpm.			
No. 1.....	304	217	110
No. 2.....	302	216	111
No. 3.....	303	217	110
No. 4.....	304	217	112
No. 5.....	304	217	111
Average.....	303.4	216.8	110.8

These average values represent those with the hypothetical "standard Stormer viscometer" discussed previously. Each of these values was plotted against its poise value and a straight line was drawn through these points, as shown in Fig. 3; this curve represents the relationship between viscosity in poises and the load in grams to give 200 rpm. on a "standard viscometer" when a hydrocarbon oil such as Bureau of Standards oil "N" is used.

Although they are not shown here, the points derived from Geddes' data<sup>2</sup> fall very close to this line. It is believed that, had Geddes had the distinct advantage of the increased accuracy of the stroboscopic timer in his work, the points would fall exactly on this line.

Krebs units, or K.U. values, have been included on the right of the graph in Fig. 3 for purpose of reference. It is estimated that the larger percentage of consistency determinations made by the paint industry are in the range of 75 to 90 K.U. It will be noted that the oil "N" at 25 C. falls just about in the center of this range. Since 25 C. is a convenient temperature at which to work, it is recommended that a hydrocarbon oil with a poise value of 9 to 10 at about this temperature be used for any calibration work. Of course, the use of Bureau of Standards oil "N" is a simple solution to the oil selection problem.

With this thought in mind, the center section of Fig. 3 was used to develop the graph shown in Fig. 4. The broken line, which falls directly down the center of the band made by the two solid lines, is a section of the "standard Stormer viscometer" line. It will be noticed that at any given poise value, the band formed by the solid lines is 8 g. wide. Therefore, this band represents a  $\pm 4$  g. tolerance from the "standard" line. This tolerance is predicated on specification paddles, accurate timing, and an oil temperature control of  $\pm 0.05$  C. With respect to timing, it is believed that accuracy obtained by the stroboscopic timer can be duplicated closely by the stop-watch method, but certainly not as easily or with as few determinations. It is recommended that the graph of Fig. 4 be used with the following method for the standardization of Krebs Modified Stormer Viscometers.

#### METHOD FOR STANDARDIZATION OF KREBS MODIFIED STORMER VISCOMETERS

The method has been divided into two sections. Part I is designed to check the free-running mechanical performance of the apparatus, and includes a check of the paddle dimensions. (An instrument

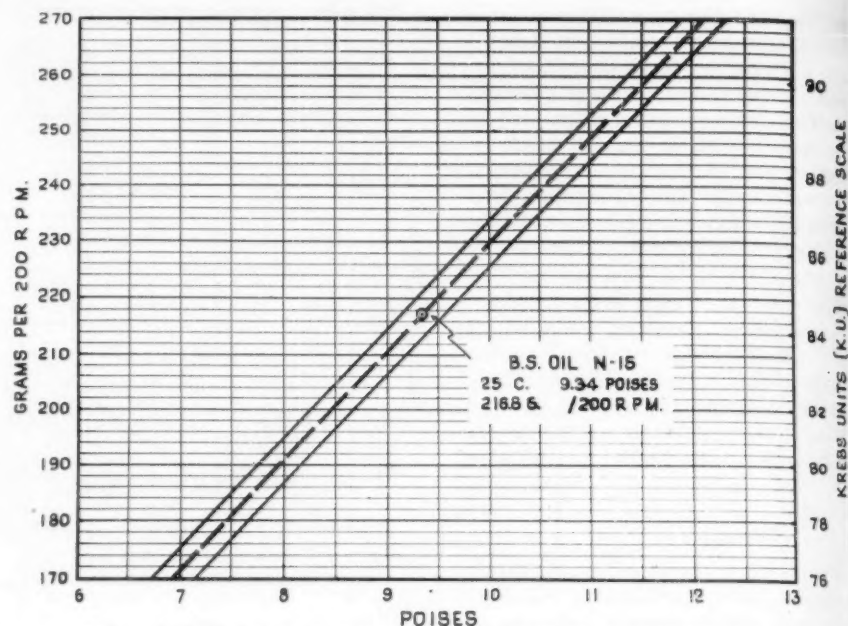


Fig. 4.—Chart Used in Standardization of Modified Stormer Viscometers.

which does not meet these requirements most certainly is not satisfactory for normal use in determining consistencies of paints.) Part II is designed to check the performance under conditions of actual use with normal loads.

#### PART I

1. Remove the paddle and weight carrier from the instrument. Make sure the string is wound evenly on the drum and does not overlap itself.

2. Hook a 5-g. weight onto the string and then release the brake. If the instrument starts to run from this dead start and continues to run through several revolutions of the string drum, it is probably satisfactory for use in normal paint work. If it does not start unaided with the 5-g. load, the instrument should be considered for reconditioning.

3. Check the dimensions of the submerged-type paddle after any foreign material has been removed. It must conform to the requirements of A.S.T.M. Specification D 562 - 47.<sup>3</sup>

#### PART II

1. Secure a sample of a hydrocarbon oil, similar to Bureau of Standards oil "N," with a known viscosity of 9.00 to 10.00 poises at 25 C. The individual worker may choose to standardize his own oil, in which case his Stormer viscometer readings should be taken at the same temperature as his viscosity determinations using the same thermometer. This procedure would tend to minimize any errors from temperature differentials.

2. The oil must be used in a quantity of no less than 1 pt. Geddes<sup>2</sup> has shown that anything less than a pint will give false readings. A standard 1-pt., round, friction top can is preferred.

3. With the specification paddle in place on the apparatus, set the oil sample in position making sure that the paddle shaft is in the center of the can.

4. Bring the oil to temperature and hold to  $\pm 0.05$  C. for at least 15 min. before taking any viscometer readings.

5. When the temperature equilibrium has been established, the weight to the nearest gram for 200 rpm. is determined. The worker is cautioned again to make sure that the string is always wound evenly on the drum and is not allowed to overlap itself at any point. The latter condition has been found to cause as much as 20 g. error in Stormer readings.

6. Using the determined gram-weight and the poise value of the oil, plot the point on the graph of Fig. 4. If the point falls within the band, the instrument under test may be considered as performing satisfactorily in comparison with the "standard Stormer viscometer"; that is, with the average value of the five new instruments used in the derivation of the graph. Of course, the method presupposes accuracy of controllable factors such as temperature, weight, timing, oil standardization, specification paddle, and operating techniques. If the plotted point falls outside of the band, such points should be checked before a decision is reached that the Stormer is faulty.

#### CONCLUSION

The method described in this paper for the standardization of Krebs Modified Stormer viscometers is believed to be reasonably simple to execute as well as sufficiently accurate for the purpose for which it was designed. It is submitted for consideration by the A.S.T.M. for adoption as a standard method.

<sup>3</sup> Method of Test for Consistency of Exterior House Paints and Enamel-Type Paints, (D 562-47), 1947 Supplement to Book of A.S.T.M. Standards, Part II, p. 183.



# Panel Discussion on "Practical Aspects of the Testing of Paint and Varnish Products"

IN VIEW of the importance of a number of details in carrying out the various familiar tests for paints and varnishes, a panel discussion on this subject was organized. The discussion took place in connection with the meeting of Committee D-1 on Paint, Varnish, Lacquer, and Related Products, held in Chicago at the Edgewater Beach Hotel, on March 2, 1949. A record of the discussion appears below:

Moderator: Francis Scofield, National Paint, Varnish and Lacquer Assn., Inc.

Panel Members: W. C. Armstrong, E. I. du Pont de Nemours & Co., Inc.; Sophus Bolme, Rinshed-Mason Co.; R. R. Bruhn, Sherwin-Williams Co.; Charles C. Hartman, National Bureau of Standards.

MR. SCOFIELD.—Gentlemen this is to be essentially an informal discussion—largely or almost entirely extemporaneous.

I have made a tentative arrangement of various test methods, that are widely used, starting out with test methods applicable to liquids, liquid coatings, the ones that are made during the drying, and finally the one on the dried film. I am going to ask members of the panel who are particularly competent to discuss them.

## LIQUIDS

One of the first liquid tests and, at least superficially, one of the simplest is the determination of weight per gallon. Practically all of us are familiar with the weight per gallon cup, but it has its peculiarities along with the rest of them. Mr. Armstrong, do you have something to say about that?

MR. ARMSTRONG.—This test, although it is really very simple, leaves plenty of room for improvement. An average determination may go something like this. You take the paint; stir it very laboriously by hand, because mechanical equipment may whip air into it; then you pour the paint into the cup. Despite your precautions, the cup gets full of bubbles and you start hammering it on the desk or some other object to get the bubbles out. Finally you get the bubbles out and put the lid on but nothing comes out of the hole in the lid because, by the time the bubbles have broken, the paint level is too low. You add more paint. This time the paint comes out the top but it also runs down the outside; the cup has to be carefully cleaned. In general, the operation is much more laborious and "messy" than you expected.

In my experience, the small cups are very seldom used for plant work because the equipment is not rugged enough to stand much abuse, and the whole operation is a little too delicate.

Furthermore, cups are generally supposed to contain 83.3 ml. (to make the weight in grams, divided by ten, give the pounds per gallon). Surprisingly enough, although this volume is accurate enough for most determinations, it is not the exact theoretical volume required.

I think there is plenty of room for improvement in this simple test.

MR. SCOFIELD.—Other tests on liquid paint are consistency tests. We have many of them. Certainly the simplest, where applicable, are the bubble tube tests. Mr. Hartman, I guess, in the laboratory, you have used them many times.

MR. HARTMAN.—I don't think that the bubble tube needs to be described to anyone here, but I do feel that there are certain precautions that should be followed when using the tubes. First, the tube to be used in measuring the test sample should be perfectly clean. It should meet the specified measurements of the tube which contains the standard oil. The air bubble should be adjusted so that it has as nearly as possible the same size as the bubble in the standard tube. And in line with making the measurement, there was a rather interesting discussion in the group meeting yesterday on viscosity as regards to the possible collection of satellite air bubbles. Any extra bubbles that tend to cling to the large bubble should be eliminated before the measurement is made.

The temperature should be watched rather carefully. You should bring the standard as well as the tube under test up to the same specified temperature. This can be accomplished more hurriedly in the water bath than in the air bath. Probably there will be some information published before long with regard to changing the term poise, which we usually employ, to stoke. Then too, there is rather positive proof, now, that we are not only measuring stokes but also surface tension.

MR. SCOFIELD.—The two other widely used liquid methods, Stormer and Mobilometer, are widely familiar. There is considerable difference of opinion as to where they fit in. If we wanted to spend most of this period, we could undoubtedly do it in discussing the details of them. It has been my experience, at least, that for most purposes the Stormer is an easier instrument to use, easier to bring the pint can of paint to the temperature of the water bath, easier to clean up the instrument, and it does not have the idiosyncrasies of the Mobilometer.

The Mobilometer has a wider range and is probably a little bit less affected by extremes of mixture and odd types of yield value and so forth, that you may get in it. Most people have their own ideas on that. The principal thing that you have to bear in mind is that with most of our pigmented products, no one number is a definition of the consistency. If they are nicely behaved paints, why, two numbers may do. Sometimes you need a whole curve and time history and you not only need to know something about the paint, but you need to know what has happened to it. So it gets quite complicated, much more complicated, incidentally, than either of those instruments can handle.

A simpler plan and definitely adequate for many purposes is the use of the various efflux cups, of which I believe one viscosity committee has collected some forty examples.

MR. BOLME, you have probably run more lacquer through these than any of the rest of us.

MR. BOLME.—Automobile lacquers are run mostly under the No. 4 cup. For that work, the No. 4 cup has become pretty well established. However, lacquers as they run now, as sold, are generally too heavy for proper running in the No. 4 cup. This cup is still good for running the lacquer after reduction. Other cups for running reduced lacquers, especially in a factory, could be designed so that they would be cheap and you would probably run one cup in each bath of lacquer and you wouldn't have to wash it up, you just leave it right in the lacquer and when you want to check the viscosity you just pull up the cup and start the stop watch.

The Ford cup does not always run true. When we are buying new Ford cups, we always find it advisable to

check them against the ones we already have.

I might mention also that in the heavy lacquers, we found the Stormer method, using the paddles instead of the cylinder, to be better than the Ford cup. And as a matter of convenience in factory control, we take the sample and an 8-oz. Dixie paper cup and bring the sample to proper temperature in hot water and then run the viscosity in that and afterward you can throw your cup away and you don't have to go to the trouble of cleaning.

MR. ARMSTRONG.—At one of these committee meetings a procedure for using a Ford cup was discussed where, instead of holding your finger on the bottom, you put a glass on the top of the cup. This holds the paint at a distinct level until you are ready to operate. At the desired time you slip it off and start the paint flow. It sounds like a convenient way to use the cup.

MR. SCOFIELD.—The glass is something else to clean up.

MR. BRUHN.—That is just the comment that we received when we tried the idea.

MR. SCOFIELD.—Mr. Bruhn, do you have something?

MR. BRUHN.—I should like to make one statement. I believe that anybody working with viscosity cups will agree that there is no particular significance to the size or shape of orifice used in viscosity cups. It is simply a matter of the adoption of one as standard.

The trouble that Mr. Bolme outlined with his Ford cup would be true for all such instruments. Wear is a very important factor with any viscosity cup, because of cleaning a relatively small orifice. Therefore, in any future specifications that are written on viscosity cups, there should be a check method included.

MEMBER.—The disadvantages of various types of viscosity cups have been pointed out, but no mention has been made of the Mobilometer. It has been our experience that this instrument is subject to just as much wear as, if not more than the average viscosity cup. The question which I should like to ask is this. From a production standpoint, what type of viscosity measuring device is fastest, easiest to clean, and simplest to use where pigmented products are involved?

MR. SCOFIELD.—Well, what do you think, Mr. Bolme?

MR. BOLME.—I haven't used the Mobilometer. We don't use it in lac-

quers at all and I have not done enough work with paste products.

MR. BRUHN.—It would depend upon the type of consistencies with which you are working. If you are running heavy consistencies, such as house paints, I think that the only instrument that is available is the Stormer viscometer or some modification of it. If you are running lower consistencies, I question the applicability of a Stormer and it would be my opinion that a viscosity cup type of device would give more accurate measurements.

MR. SCOFIELD.—One rather important feature of most of our pigmented liquid coatings, because they are used to change the surface appearance of objects, is their hiding power. To measure it, we vary all the way from extremely simple methods to ones depending upon complex, long formulas involving higher mathematics. Probably the simplest method we have uses the Cryptometer. Mr. Bolme, you have had some experience with that.

MR. BOLME.—We use the Cryptometer in controlling all our factory batches of automobile lacquers. We have discarded the old Cryptometer, with its V-trough, and are now using the newer type with about a one-half black and one-half white glass base. For light colors, like creams, and white, we run on a black scale and this makes it easier. All the other colors we run on a white scale. The reading seems to have the same meaning in each case. We find this quite useful in controlling our production and as an indication of performance expectancy. For instance, on a 0.00035 Cryptometer, we find that with the reading above 25 there will probably be an advantage in using a ground coat under the lacquer. On full lacquers, like certain maroons that have very important hiding power, we use 0.0007.

MEMBER.—I should like to comment briefly on the Cryptometer because I think there is a serious problem that arises with its use. The problem is that our Cryptometers wear out too rapidly in use, particularly when used for production control.

MR. SCOFIELD.—The other method that is widely used is the brushout method. I think most of us have had our adventures with it and am not sure all of us want to remember them if we can help it. Mr. Bruhn, perhaps you can tell us something about the brushout method.

MR. BRUHN.—You describe it as a method that is used widely for evaluating wet hiding. We like to think of the

brushout method as a practical control comparison of two paints of similar nature, and our brushout tests are of two rather widely divergent types. In one test, we make a brushout on an area of approximately 4 ft. square. The background is white except for a 4-in. gray stripe across the center. We consider this gray-to-white contrast as representing the optimum that the user could expect to obliterate in a one-coat application of a finish. It is strictly a practical test in that the painter applies the paint at the natural spreading rate for that product.

The second test, a little more exact, but still only a comparison, is the paint-out by weight-difference on Morest charts, and for that method we use a standard paint, at various spreading rates, and compare it against paint-outs of the test paint; by bracketing the sample with standards we describe its opacity.

Most of our brushout tests are over gray-white surfaces rather than over black-white, because it is very seldom that you get reasonable one-coat hiding over a black-white contrast, especially with white paints.

MR. SCOFIELD.—When the dry hiding power is measured, of course, you get much higher precision, assuming that you can control everything, by measuring the contrast ratio over black-white, gray-white, or black and gray. I think these backgrounds have all been suggested.

MR. ARMSTRONG, don't you have some experience on contrast ratios?

MR. ARMSTRONG.—Well, of course, contrast ratio is not a complete method of running hiding tests, it is a method of expressing opacity under certain conditions of film thickness and I think it is, by far, the best way of expressing opacity that we have. I think it is here to stay. One of the present troubles is that very few people really understand the significance of the numerical values. Generally they are much inclined to underestimate the differences. For example, in an actual case one paint had a 0.98 contrast ratio and another had 0.97. You think, "Well, there is little difference." But actually when you build up the opacity of the second paint to 0.98, you find you have increased the paint's cost approximately 50 cents per gallon to correct a seemingly small deficiency in opacity.

Another illustration of what I mean is that very often you see literature stating that surfaces having a ratio of 0.98 are about visually equivalent. This has not been our experience at all. We have found two surfaces to appear



similar only when they have a ratio of approximately 0.995 to 0.998.

MR. SCOFIELD.—This has been my observation. Is there anything further? If not, we shall now discuss Fineness of Grind.

In the past few years there has been much publicity on it; in fact, at one of the Federation conventions, there were several papers presented on Hegman gages which are used to determine grind requirements. Mr. Bruhn, you have had experience with these gages.

MR. BRUHN.—The Hegman gage is well publicized. I think you all are acquainted with description of the basic gage. It usually consists of a wedge-shaped slot which has been machined into a steel block which is exactly 5 in. in length. The slot varies in depth from zero at one end to four mils at the other. The Hegman gage, as it is popularly known in the trade, has a  $\frac{1}{2}$ -in. slot or wedge divided into eight divisions which correspond to the North Grind Standards. The New York Production Club has set up the Production Club Scale which uses the same wedge but the scale is in ten divisions.

I would make one suggestion to people working with the Hegman gage which I think they will find quite useful. Start using the wedge depth in mils rather than in any of the arbitrary scales. You will find remarkable correlation between the wedge depth in mils and your grind requirement for the product which you are formulating. The Hegman gage user should be cautious about several points. One of the most important concerns the method and the degree of dilution of the sample for grind determination. The sample should be reduced as nearly as possible in the manner that it will be reduced for final usage; if you have a paint that is reduced in the thin-down tank with additional oil, and very slow solvent, follow that method to reduce your grind determination sample.

Another point is that you will have to decide where to read your end point. Our institution has used the Hegman gage as a standard for several years and we have just set up an advisory committee which will study the interpretation of the gage and will instruct the personnel of our various factories in the correct reading of the gage. For reproducible readings between individuals, some graphic standards are indicated.

Mr. Armstrong, in discussing this problem yesterday, brought up this point. On the Hegman gage it is quite simple to identify the type of grinding equipment used in preparation of a given paint. You will note that the ball mill or pebble mill batches will "break"

on the Hegman gage at a sharp point. The roller mill paste will be sharp also but if you run stone mill paste you will find scattered particles all the length of the scale.

MR. SCOFIELD.—Mr. Bolme, do you have a question?

MR. BOLME.—Our control chemist told me that a Hegman gage should be read within 10 sec. after you run the steel block over the case. Has Mr. Bruhn any comment on that?

MR. BRUHN.—The time would depend upon the solvents used in the paint. When testing products diluted with mineral spirits, we have waited as long as 30 sec.; however, products diluted with faster thinners, some of them lacquer types, for example, will dry in a matter of a very few seconds, especially in a draft. For these products, the readings must be made immediately. We found that the average time of reading for the normal operator is very close to 6 or 7 sec. We have timed a great number of operators and found that they don't vary. The time that it takes to make the draw-down, pick up the gage, hold it at the angle for viewing is at a practical average of 6 to 7 sec. Regarding the angle of viewing, I think that all of us are more or less inclined to look at the gage at a grazing angle facing a bright source with surrounding shadow, for example, looking at the window from the center of a room. That does not seem to be of great importance except that it enables the operator to observe the shadow cast by the particle rather than having to resolve the particle itself.

MR. ARMSTRONG.—I have examined many paints having consistencies apparently of a fairly high yield value and in which the liquid didn't run away from the particles, letting them stand out sharply on the gage. In such paints, a small amount of added blown oil tended to knock down the false body, but once or twice I have examined paints in which blown oil is not effective and they were very difficult to read. I don't know what the answer is for those paints.

MEMBER.—How much would it cost to make the Hegman gage with a trough, 1 or  $1\frac{1}{2}$  in. wide, in order to get the same effect Mr. Bolme mentioned, that you get with the glass and spatula?

MR. BRUHN.—I don't know about 1 or  $1\frac{1}{2}$  in. but we have a number of gages in our plant now that are 2 in. wide and the price for one gage is \$90.

MEMBER.—Mr. Chairman, on the subject of drawing down the paint for fineness of grind, I have heard differ-

ences of opinion as to the angle of the blade in drawing down the paint in the channel. Can any of the men here comment on that?

MR. BRUHN.—The angle of the blade, in our experience, does not make any particular difference as long as you remain within the top 40 deg. out of 180 deg. In other words, any reasonable angle is satisfactory. Another point is that any normal speed is suitable; it must be slow enough that paint will enter the gage. The speed can be varied also.

MR. SCOFIELD.—Regarding that angle I think it is well to bear in mind that the draw-down blade is subject to unexpected wear. It is likely to wear more if you routinely draw it at an angle than if you habitually draw it vertically.

MR. BRUHN.—The blade should be checked frequently. One of our departments claimed their gage gave readings one division different from all other departments'. It was found that the scraper bar was so worn on the ends that if one end was pressed tightly against a flat surface, the other remained 0.005 in. from the flat surface and acted as a "rocker." The convex blade depressed the paint in the center of the trough.

MR. SCOFIELD.—Of course, this condition is more serious with a wide channel than it is with a standard one.

MR. BRUHN.—That was a wide standard gage.

MR. SCOFIELD.—Mr. Bolme, you have done some work using another method, I believe.

MR. BOLME.—Testing a paint with a knife on a glass is an old method used by painters for a long time. It is not scientific; that is, you cannot put it down in figures; you cannot put it into any specifications. But it has certain advantages. Speed is probably one attribute and you can carry just your knife and glass around the mills and make tests as they grind.

To prepare the knife used in this method, secure a small spatula and with a fine hone grind down all the sharp edges and burrs and grind the face of the spatula to a slight convex. In the case of a fine-ground paint, a slightly larger area results when the knife is used on the glass. You view the paint as you smear it out and I find this technique makes it easier to distinguish between the pigments of the coarse grind and those that are picked up accidentally while taking the sample or otherwise. For a paint having upper and lower limits to fineness, such as an undercoat where shortness or poor adhesion may

occur, I find a Hegman gage is preferable to the knife and glass method. But when the fineness is the objective that you paint, such as an automobile lacquer or a fine enamel, I have found the knife and glass to be fast and entirely satisfactory.

MR. SCOFIELD.—There are a number of other tests that are performed on liquids but most of them are either fairly well known or not particularly significant, so I think we will now discuss another important topic.

#### DRYING TESTS

Since paint is a drying type of material, one of the obviously important factors is drying time. It has been discussed at great length by various groups of this organization and the conventional methods of touching the film with your finger are well known. The obvious defects also are well known. The agreement reached by cooperative tests has been poor in most cases, but that is almost entirely, especially in the early phases of the drying of the film, a matter of precise control of conditions. I think most of us realize that temperature must be controlled closely to get reproducible drying-time results. I am not sure that we all are aware of the significance of air circulation in drying-time tests. All who have run a draft test know how much faster a varnish dries in a draft than it does in relatively still air, but I have been startled on occasions to notice the difference in drying time between a panel on the front edge of the desk where there is some movement of air and one which is back against the wall where the air is relatively still. I think that failure to control air circulation frequently accounts for some of our gross discrepancies.

In recent years, some of them not so recent, a number of mechanical devices have been used. Mr. Bruhn has had experience with several and he can briefly outline some of their characteristics.

MR. BRUHN.—All of you undoubtedly recently have received literature from Reichhold Chemical Inc. (RCI) on their drying recorder. Probably many of you have one in your laboratory and are trying to work out a way to use it. We have been working intensively with the RCI drying recorder in our laboratory for the last two months and have learned interesting facts about the instrument.

The recorder is essentially a clock which times a slow pull of its stylus across a paint film. The first thing of importance that we found was that one must reproduce paint film thicknesses very accurately to get reproducible drying rate tests.

The second fact that we found was

that, in addition to control of temperature and humidity, lighting conditions must be reproduced very exactly. You must test either in the absence of light or under artificial illuminant. We accomplished this by putting our recorder directly under a fluorescent unit, leaving the light on during the drying tests. By controlling temperature, humidity, air circulating, and illumination we obtain almost perfect duplication of results.

We have altered the basic RCI design by changing the stylus from a small hemisphere needle  $\frac{1}{32}$  in. in diameter, to a blunt "hoe"  $\frac{1}{4}$  in. across. We did this in order to observe the edges of the drying track without having to look down into a very tiny "valley." We have an end point, or, more accurately, a length of the stylus traverse, which we can correlate closely with manual set-to-touch time and the location of this point is independent of the stylus weight.

The second point which is some measure of thorough drying is the point where the stylus no longer penetrates but rides on top of the paint film. This is dependent upon the stylus weight.

I would object to the Sanderson type of drying recorder on the basis that the pile of sand remains on the paint film until the test is complete. If the substrata of the paint are plastic, a slow indentation due to the weight of the sand particles occurs; consequently, the instantaneous drying time is not measured. The time which is measured is an interval which depends not only on the paint composition but on the weight of the sand.

MR. SCOFIELD.—Thank you Mr. Bruhn.

Two other tests that concern the drying of varnishes are the gas test and draft test. They were well established years ago. Mr. Hartman, have you anything to say about them?

MR. HARTMAN.—Not much. I might comment briefly on them. Of course, I think we know that for quite some time, the gas test has been considered more or less an indication of the proper cooking of the varnish. There is one thing that, as far as I know, has never appeared in the specifications covering procedures for making the gas test. It is quite important that the test be made as soon as possible after the set-to-touch time has been determined. If you wait until the next day or until the afternoon of the same day, sometimes you may get variations in the results of the test. Under extreme air conditions of the laboratory, you may get as much as 15 min. variation in set-to-touch time, which may affect the results of the gas test.

Your time of drying of the different test films that are prepared during the interim is one-fifth of the set-to-touch time; that is, if the film sets to touch in 30 min. the test films will have dried 6, 12, 18, and 24 min. before exposure. Since the dry-time periods for the specimen films vary with surrounding air conditions, if you don't make your test shortly after you determine the set-to-touch time, you may possibly run into difficulty. You may have only one of the test films that will show "crow's footing," frosting, or something of that nature, whereas if you don't test until the next day, you may have two or three of the test films that show frosting or crow's footing.

In the draft test, the panel is usually placed in a vertical position. This perhaps is not the most desirable position to have the panel while the air is being blasted against it because there is a much thicker film over the bottom part of the panel than there is over the top half and, if the analyst is not careful, he may misjudge the qualities of the varnish. In judging the draft test properties of the varnish, only the upper half of the panel should be considered since that portion probably is more representative of the thickness of the coating put on in practice.

There is a modification of this method, in which the panel is laid in a horizontal position and the fan tilted so as to get the same angle of draft against it. This may eventually replace the old method as it permits the testing of films of uniform thickness.

MR. WAYNE FULLER.<sup>1</sup>—Mr. Chairman, we have had an RCI dry recorder and have discovered several disadvantages.

One, for instance, is that when you change the stylus, you get a result which is variable.

MR. BRUHN.—You do, to some extent. In our work we were not interested in that point. We have made our equipment convertible so we can interchange styluses.

MR. FULLER.—Do you lose the ability of the stylus to ride up on top of the surface after you once get drying?

MR. BRUHN.—No. It comes up very definitely.

MR. FULLER.—Apparently a worthwhile modification with this instrument would be the use of steel panels (on which the paints are applied) wide enough for the operator to apply paints with a doctor blade. This we are going to do and I think it will be a most useful variation to control film thickness.

<sup>1</sup> Director of Research, Grand Rapids Varnish Corp., 1350 Steele Ave., S. W., Grand Rapids 2, Mich.



Mr. BRUHN.—We use glass plates and have made a new type draw-down gage with three adjacent sections for three draw-downs, each section  $1\frac{1}{4}$  in. wide so all three sections will fit the Reichhold Chemicals Co., dry recorder.

#### DRY FILM TESTS

Mr. SCOFIELD.—When we obtain films we make two visual judgments, color and gloss. When we make these decisions, there are two principal determinants: (1) preparation of the panel (about which a good deal of care has to be exercised, but about which all you can say is to do it the same way every time) and (2) the conditions of illumination and view. In the case of gloss, it is mostly a question of angle; in the case of color, it is both angle and type of illumination. Some day we hope the paint industry will have a standard luminant. We need it so the buyer and seller can agree. The industry has a number of instrumental methods. Time does not permit lengthy discussions of the instrumental methods for the measurement of color. They are diverse and each of them has its tricks, both in measurement and interpretation of results. The Gardner-Hunter instrument is widely used. Its idiosyncrasies have been discussed at great length. One group discussed the measurement of gloss yesterday but those discussions cannot easily be summarized.

The Photovolt Corporation has made a gloss meter. Mr. Bruhn, I think you have used it. Could you tell us what you think of it?

Mr. BRUHN.—The Photovolt gloss meter is convenient from the standpoint that you have a direct reading meter. It is semiportable.

One point that we have noticed is the difficulty of attempting standardization of the Photovolt gloss meter by means of porcelain standards; a series of porcelain standards compared on eight photovolt instruments showed each instrument to be different. Mr. Hammond is at our plant this afternoon trying to eliminate this trouble.

Mr. SCOFIELD.—Mr. Armstrong, you have used the Aminco-Scott instrument.

Mr. ARMSTRONG.—We have used it under various conditions. It is a good instrument. You can vary the angles of illumination and reception. It gives pretty good results on high gloss panels; that is, it usually checks visual ratings. In practice, the degree of correlation may be somewhat as follows: If you take ten panels and rate them visually, the machine rating will probably check the visual ratings. If you take twenty panels, you may find one meter reading out of line. An unknown peculiarity of some surfaces makes the meter go astray. However, it gives closer checks than other instruments which we have tried. I don't believe that you can buy an Aminco-Scott today. Although it is a good instrument, it does have some bad features.

Mr. SCOFIELD.—A semivisual method was introduced some years ago by the

Detroit Production Club. Mr. Bolme, you have used that, I think.

Mr. BOLME.—The advantage of that method is that it measures what the eye sees; that is, the reflection of some object in the surface of the finish is seen. The difficulty we experienced has been in the preparation of the graduated gloss panels. More work should be done on that. I have had little time to follow this problem.

Another advantage of this last meter is that it is entirely independent of color. The meter, as you may know, is encased. The lid is ground glass, with black cross bars drawn on it, and the reflection of these black cross bars in the glass standards, compared to the standard measured, gives you the gloss. The preparation of the standard is still the stumbling block, but if we could find some means of measuring gloss by actually viewing it with the human eye instead of the electric eye, we would probably get more nearly a measure of what a person wants in a paint rather than what a machine wants.

Mr. SCOFIELD.—Well, gentlemen, I am sorry to say our time has expired. There are many more test methods we haven't covered. Maybe, if there is sufficient demand, the Program Committee will ask us to convene again sometime.

I wish to thank the members of the panel and the audience.

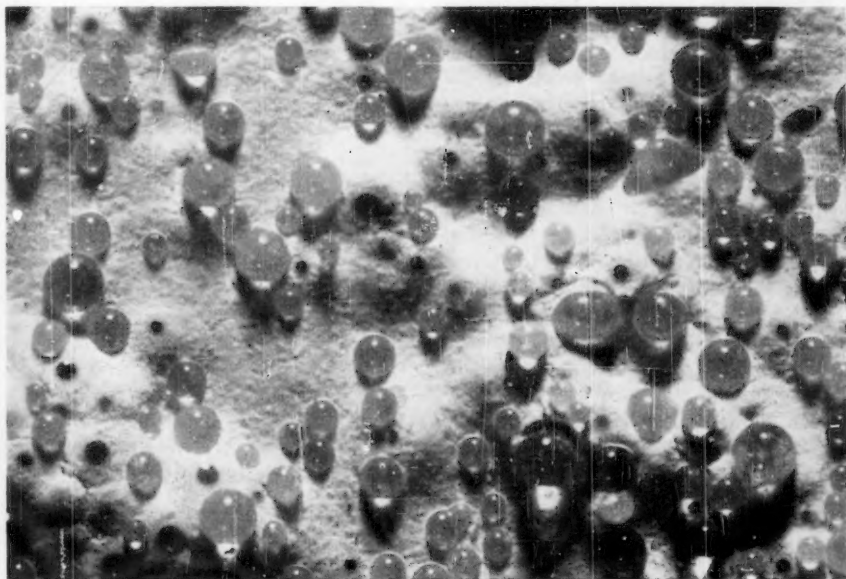
#### Use of A.S.T.M. Standards

AN INTERESTING article in the journal *Westinghouse Engineer* describes Thermoset varnish, calling varnish "The First Line of Defense." The article points out that varnish, the last layer of insulation on electrical equipment, is really the first line of defense against heat, moisture, etc. A failure in the varnish film will almost certainly lead to damage. A table in the article gives the properties of Thermoset varnishes, the various properties being determined in accordance with A.S.T.M. standardized procedures.

#### Errata—Paper on Impact Resistance of Plastics

IN THE paper by H. M. Quackenbos, Jr., J. M. Hill, Jr., and C. E. Staff, "The Significance of the Charpy and Flexure Tests in Evaluating the Impact Resistance of Plastics," July *Bulletin*, p. 57, the author wishes to call attention to two of the symbols appearing in Eq. 1 which were described incorrectly. Properly described, they become:

- $w$  = circular frequency, radians per second =  $2\pi \times$  frequency in cycles per second  
 $u$  = mass per unit length divided by the gravitational acceleration, g.



"Weathered Traffic Paint"

First prize-winning photograph, Electron Micrograph and Photomicrographs Section, Photomicrographs Group, (macrographs of particles) in the Sixth A.S.T.M. Photographic Exhibit, by E. J. Thomas, American Cyanamid Research Laboratory. (70X)

# Impact Testing of Plastics: Elimination of the Toss Factor<sup>\*1</sup>

By Bryce Maxwell<sup>2</sup> and L. F. Rahm<sup>2</sup>

## SYNOPSIS

Presented here is a new method of measuring the impact strength of polymeric materials which eliminates some of the extraneous energies measured by the standard Izod test, the most important of which is the energy to toss the broken half of the sample. The apparatus is described and the data for several materials both by the new method and the standard Izod method are tabulated. Comparison of results show that this new method ranks materials in the proper order as indicated by service performance of the materials. Experiments are described which lead to the conclusion that the impact strength as determined by this new method is independent of the mass of the broken sample. This permits testing over a wide range of velocities to which this method is easily adapted.

IN ORDER to synthesize a plastic with high impact strength it is first necessary to have a basic understanding of the property of impact resistance. To obtain this knowledge it is essential to be able to measure impact strength without the inclusion of extraneous factors, over a wide range of velocities. From an investigation of the energy that various polymeric structures can absorb and the manner in which this quantity of energy varies with the velocity of impact and with the temperature, some understanding of the phenomena of impact strength should be found.

The errors inherent in the standard Izod test (D 256 - 47 T)<sup>3</sup> have been pointed out by Telfair and Nason (1)<sup>4</sup> and others. Certain additional or extraneous energies are included in the Izod reading, and only one testing velocity can be conveniently used with the standard machine. The lack of correlation between Izod impact strength and the impact performance of components in service has been pointed out by several writers. The most outstanding inconsistency in this respect as pointed out by Hazen (2) is between wood flour and mica filled phenolics;

the mica filled is stronger in the standard Izod test but the wood flour filled is more impact resistant in service.

Telfair and Nason point out that the Izod reading includes the energy to initiate and propagate the fracture, energy to deform the specimen plastically, energy to throw the broken end of the specimen, and energy lost in vibration of the machine. All these energies except the first are extraneous and should not be included in the impact strength. Burns and Werring (3) and Zinzow (4) have suggested that the energy to toss the broken sample, commonly called the "Toss Factor," may be a large part of the energy measured by the standard test. Assuming that the velocity at which the sample is tossed is a constant, the energy to toss will depend on the density of the sample. Therefore heavier material will have higher toss factors. This will lead to relatively increased Izod impact strength readings for dense materials. If the test could be made at an increased velocity in an Izod type machine, the toss factor for heavy materials would be increased to a greater extent than for lighter materials.

Attempts have been made to devise testing methods which will not include this added energy. The machines of Telfair and Nason (1), Stock (5) and Callendar (6) have attempted to do this by hitting the sample with a blow of just enough energy to either crack or break the sample, that is by reducing the toss of the specimen to zero or to a small value which can be measured. These machines require a large number of specimens to determine the reading, and they do not permit the testing of materials at a constant velocity; that is, there is a very large deceleration of the striking member during the time the sample is being broken.

The machine described in this article is capable of testing samples at various velocities and is believed to be free from the toss factor problem.

## BASIC THEORY

In the standard pendulum machines and also in the Guillery flywheel machine, prior to the impact, the specimen is stationary. After the impact, the broken piece is moving at some unknown velocity. This kinetic energy in the broken sample must be supplied by the striking member, either the pendulum or flywheel. If the total energy removed from the striking member is called the impact strength, it will be in error by the amount of kinetic energy in the broken sample. On the other hand if, prior to impact, the specimen is in motion, it will contain the kinetic energy to toss itself after it rebounds from an anvil, and this energy will not appear as a reduction of the angular velocity of the pendulum or flywheel. The machine described in this paper operates on this principle.

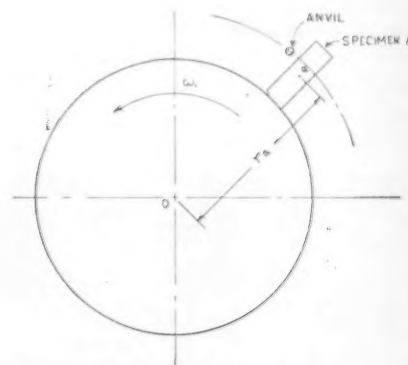


Fig. 1.—Basic Principle of Operation.

If a standard Izod specimen is attached to the periphery of a flywheel as shown in Fig. 1, the energy in the rotating parts prior to impact is the sum of the energy in the flywheel and that part of the specimen which is not broken off plus the energy in the protruding part of the specimen, A, which is to be broken off. This can be expressed by:

$$E_1 = \frac{1}{2}I\omega_1^2 + \frac{1}{2}M_A r_a^2 \omega_1^2 + \frac{1}{2}M_A \omega_1^2 \dots (1)$$

where:

$E_1$  = energy in rotating parts prior to impact,

$I$  = moment of inertia about center,  $O$ , of flywheel and that part of the sample not broken off,

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<sup>1</sup> The work described in this paper has been done under the sponsorship of the United States Army Signal Corps, and Navy: Bureau of Ships, Bureau of Aeronautics, Bureau of Ordnance, and Office of Naval Research, under Contract No. W-36-039-sc-32011.

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<sup>3</sup> Tentative Method of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 - 47 T), 1947 Supplement to Book of A.S.T.M. Standards, Part III-B, p. 50.

<sup>4</sup> The bold face numbers in parentheses refer to the list of references appended to this paper.



$\omega$  = angular velocity prior to impact,  
 $M_A$  = mass of protruding sample,  
 $a$  = radius from  $O$  to  $a$ , the center of gravity of  $A$ , and  
 $I_a$  = moment of inertia of  $A$  about its center of gravity,  $a$ .

Call  $E_2$  the sum of all the energies after break, neglecting the spinning of the broken sample. (An estimate of the energy dissipated in the spinning indicates that it is negligible.)  $E_2$  equals the sum of the energy in the flywheel plus the energy in the broken specimen,  $A$ , plus the energy to break plus the energy dissipated in the nonelastic impact of the specimen and anvil.

That is:

$$E_2 = \frac{1}{2}I\omega_2^2 + \frac{1}{2}M_A r_a^2 \omega_2^2 + E_B + E_p \quad (2)$$

where:

$\omega_2$  = angular velocity of flywheel after the break,  
 $r_a \omega_2$  = linear velocity at which broken specimen rebounds from anvil,  
 $E_B$  = energy to break, and  
 $E_p$  = energy of plastic impact between sample and anvil.

Conservation of energy dictates that  $E_1$  must equal  $E_2$ . Equating Eqs. 1 and 2 and solving for  $E_B$ , the energy of break:

$$E_B = \frac{1}{2}I(\omega_1^2 - \omega_2^2) + \frac{1}{2}M_A r_a^2 \omega_1^2 - \frac{1}{2}M_A r_a^2 \omega_2^2 - E_p + \frac{1}{2}I_a \omega_1^2 \quad (3)$$

The energy dissipated,  $E_p$ , in plastic impact between the sample and the anvil should not be included as part of the impact strength of the material. To do so would be prejudicial to elastic materials and would also mean that we were testing for only one specific condition of impact. Therefore for any condition of impact between specimen and anvil, from completely elastic to completely plastic:

$$\frac{1}{2}M_A r_a^2 \omega_2^2 + E_p = \frac{1}{2}M_A r_a^2 \omega_1^2 \quad (4)$$

That is, the sample will rebound with the same velocity with which it struck the anvil.

Substituting this in Eq. 3:

$$E_B = \frac{1}{2}I(\omega_1^2 - \omega_2^2) + \frac{1}{2}M_A r_a^2 \omega_1^2 - \frac{1}{2}M_A r_a^2 \omega_1^2 + \frac{1}{2}I_a \omega_1^2 \quad (5)$$

Obviously the energy in the broken specimen,  $A$ , prior to impact cancels the energy in the broken part after the break.

Therefore the impact strength of the material is given by:

$$E_B = \frac{1}{2}I(\omega_1^2 - \omega_2^2) + \frac{1}{2}I_a \omega_1^2 \quad (6)$$

In an actual test the  $\frac{1}{2}I_a \omega_1^2$  factor can be neglected, as it is approximately 0.0002 ft.-lb. and therefore negligible.

From Eq. 6 the impact strength of any material can be found since  $I$  is known and  $\omega_1$  and  $\omega_2$  can be measured.

## APPARATUS

The actual apparatus used is shown schematically in Fig. 2. It was found necessary to reduce the moment of inertia of the rotating member to a minimum in order that a measurable change of angular velocity would take place when the sample was broken. This was accomplished by making the flywheel

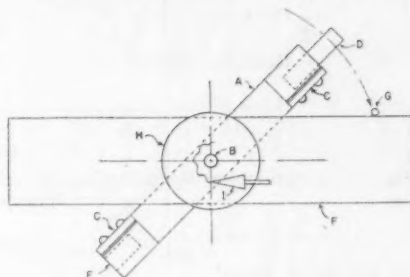


Fig. 2.—Schematic Drawing of Impact Testing Apparatus.

nothing more than an aluminum bar,  $A$ , 18 by 2 by  $\frac{1}{4}$  in., with a  $\frac{3}{8}$ -in. steel shaft,  $B$ , through its center. At both ends of the bar there is a clamping device,  $C$ , for holding a standard Izod specimen. In a test, the specimen to be broken,  $D$ , is placed in one end and one half a specimen of the same material,  $E$ , is placed in the other end. With this arrangement the rotor is dynamically balanced after the impact has occurred. The shaft is supported in ball bearings in the center of the frame,  $F$ . Brass weights can be attached to the rotor to increase the moment of inertia for testing the stronger materials. The movable anvil,  $G$ , is attached to the frame near one end so that when in position it can hit the sample at 0.866 in. from the edge of the gripping device.

The angular velocity is measured by a smoked disk,  $H$ , and vibrating pointer,  $I$ . The pointer vibrates at a known frequency, tracing a spiral sine wave on the smoked disk. During the test the pointer is moved sideways along a radial line so that the spiral sine wave it traces gives a complete record of the velocity of the rotor both before, during, and after the impact, without overlap. It is realized that this is a simple but laborious way of measuring angular velocity; nevertheless it is sufficient for a research machine. In a similar machine to be used for routine testing it would be advisable to use a more convenient method such as an electronic timer.

It was deemed inadvisable to attempt to calculate the moment of inertia of the rotor due to its irregular shape. A much more accurate determination can be made by measurement using the method shown in Fig. 3. The rotor,  $A$ , including the timing disk but not the

specimen, was rigidly suspended from a torsion pendulum and the period of oscillation of this pendulum was accurately determined. Next, a bar,  $B$ , of known moment of inertia was attached to the rotor and period of the compound torsional pendulum determined. The moment of inertia of the rotor could then be found from:

$$I_r = \frac{I_k}{\left(\frac{f_r^2}{f_2^2} - 1\right)} \quad (7)$$

where:

$I_r$  = moment of inertia of rotor,  
 $I_k$  = known moment of inertia of added bar,  
 $f_r$  = frequency of rotor alone, and  
 $f_2$  = frequency of rotor and known bar.

In an actual test of impact strength the sample is mounted in the rotor so that the center line of the notch is at the face of the grip. The rotor is brought up to the desired testing velocity and allowed to run free. The timing device is started and as the pointer moves out it trips the anvil into the path of the sample. After the break the rotor is brought to a stop and the timing disk removed. By measuring the degrees per cycle of the pointer before and after the break,  $\omega_1$  and  $\omega_2$  can be determined from a simple nomograph. An allowance for windage and friction may be found from measurement of the loss in velocity for one complete revolution running free. The contribution to  $I$  of the two half samples remaining with the rotor may be found from a chart of moment of inertia *versus* density. This facilitates the calculation of  $I$  for various materials. Knowing  $I$  and having measured  $\omega_1$  and  $\omega_2$  the impact strength can readily be found from Eq. 6. The result is expressed as foot pounds per inch of notch for comparison with the Izod results.

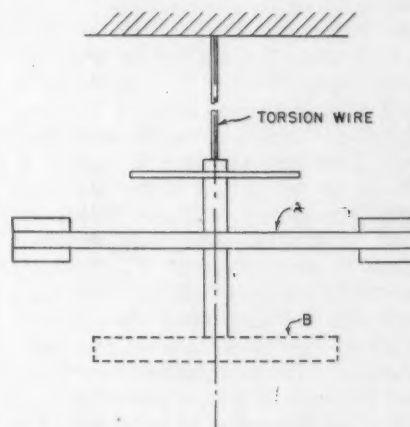


Fig. 3.—Apparatus for Determining the Moment of Inertia of Rotor.

## RESULTS

Ten standard Izod type specimens of each of nine materials were prepared. The thermosetting materials were compression molded at the optimum molding conditions. In the case of the thermoplastic materials, the specimens were machined from  $\frac{1}{2}$ -in. thick cast sheets. The regulation notch was milled into each sample in the prescribed manner.

Five specimens of each material were tested in the new machine at a velocity of 11 ft. per sec. Reproducibility of results was found to be comparable to standard impact tests. The other five specimens were tested in a standard Izod machine. All tests were performed at room temperature.

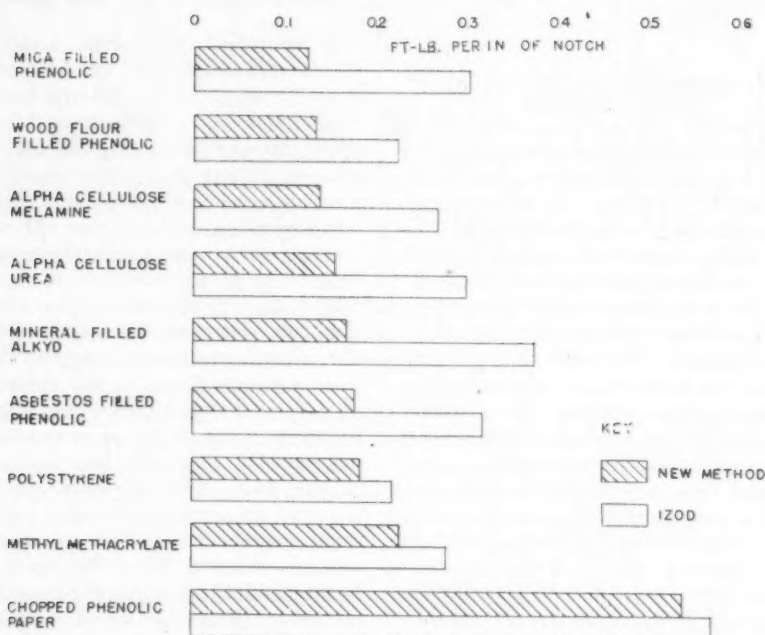


Fig. 4.—Comparison of Izod Impact Strength with Results of Tests by New Method.

The results of these studies are shown in Fig. 4 in the form of a bar graph arranged in order of increasing impact strength. The difference between the impact strength as determined by the two methods is the toss factor. This means that the energy to toss the broken sample varies from about 0.025 to 0.205 ft.-lb. per inch of notch for these materials, or in some cases more than half of the observed Izod strength. It should be noted that if the materials were arranged in order of increasing Izod impact strength, the sequence would be very different. For example, one would conclude from the Izod readings that the mica filled phenolic was much stronger than the wood flour filled phenolic. The new method rates these two materials in the reverse order, thus correcting the rating to agree with the service performance usually observed.

In order to insure that this new

method of testing is not affected by the weight of the sample—that is, to insure that the toss factor is not involved—the following experiment was performed. The mass of two standard samples of polystyrene was increased by attaching a 50-g. brass weight to each at its center of impact. One of these composite samples was tested in the Izod machine and indicated an impact strength of 1.52 ft.-lb. per inch of notch or an increase of sevenfold over the standard Izod value. The other composite sample was tested in the new machine and indicated an impact strength of 0.63 ft.-lb. per inch of notch. This is an increase of 0.447 ft.-lb. over the result for standard polystyrene specimens in the new machine. This relatively slight increase can be accounted for by the spinning of the specimen. In the conventional test in the new machine, this spinning was neg-

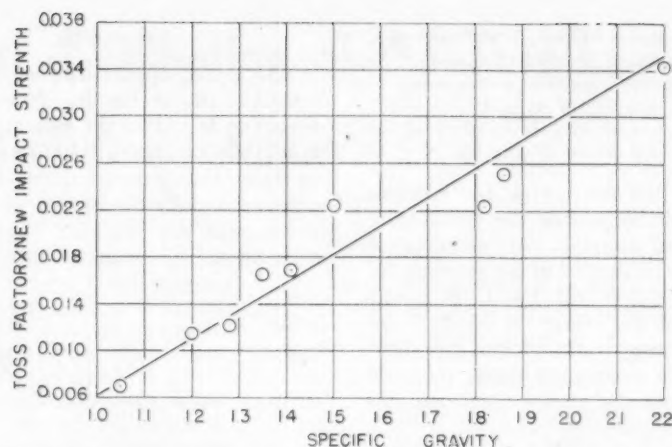


Fig. 5.—Relationship Specific Gravity and Product of Toss Factor X New Method Impact Strength.

lected because it was found to be of the order of 0.02 ft.-lb. and practically constant for all materials, but with the composite specimen, estimation of the spin gives 0.54 ft.-lb. This is a good approximation of the observed energy increase, considering the difficulty of estimating the spin energy, and we may conclude that the new method of testing is free of toss factor.

It is generally agreed that the toss factor in the Izod machine varies directly with the density of the material. It would also seem probable that the energy to toss would be proportional to the velocity at which the broken sample was thrown. This velocity in turn should be inversely proportional to the toss-factor-free impact strength. If the data of Fig. 4 are plotted as toss factor times new method impact strength versus the specific gravity of the material as shown in Fig. 5, the following straight line relationship is found:

$$(\text{Toss factor}) (m) = \frac{\text{specific gravity}}{\text{new method impact strength}} - n$$

where  $m$  and  $n$  are constants, thus confirming the above theory.

It would seem reasonable from the above that the standard Izod impact strength could be corrected for the toss factor. This could be done if the velocity at which the sample is thrown could be found. Unfortunately no convenient method for doing this is available.

Samples of various materials have also been tested at velocities ranging from 7 ft. per sec. to 50 ft. per sec. No marked trend with velocity has been found. On careful analysis it seems reasonable, considering the stress concentration in the notch as described by Morey (7), that low energy breaks should occur in all the materials tried. For this reason it is planned to redesign the testing machine for use with a tension type sample in which the velocity effect can be more readily observed.



## CONCLUSIONS

From these studies we can reach the following conclusions:

1. The new method of measuring impact strength described herein gives energies of impact strength that do not include the toss factor or energy to throw the broken sample, and that tests of impact strength of plastics can now be performed at a wide range of velocities.

2. The toss factor in the Izod readings depends directly on the density of the materials and inversely on the true impact strength. In some cases the

toss factor is more than half the observed Izod reading. A correction in the Izod reading cannot be made since the velocity of throw cannot conveniently be measured.

3. Impact tests by the new method give results that rate the various materials in an order of relative impact strength which seem to agree with the observed impact resistance of the materials in service.

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## DISCUSSION

MR. W. A. ZINZOW.<sup>1</sup>—I believe this paper as presented makes an appreciable contribution to the art of impact evaluations. The authors are to be congratulated on the results of the work that is presented in this paper.

There are a few points with reference to the paper which have been discussed in private with one of the authors before the meeting. I would like to repeat these comments here. I believe the paper would be of slightly more value to those who may want to do something with it, if a few more details were given particularly with reference to the method of bringing the rotating member up to speed and the mechanism used for releasing it before the instant of impact. I am quite sure the authors will be able to supply this information to those interested but, generally, if such details are included in such a paper it is helpful.

It is hoped that the publication of this question will help clear up questions about the impact test which have been the subject of much speculation in the past. We all appreciate very much the difficulties in the way of impact testing and particularly the interpretation of the test results. Information presented here will, no doubt, help to clear up some of the points involved.

MR. DAVID TELFAIR<sup>2</sup> (by letter).—The method of measuring impact strength of plastics as described by Maxwell and Rahm suggests the possibility of a simple and rapid test which is free from "broken end error." Such a test would be particularly advantageous

in the testing of relatively brittle materials.

I should like to raise two questions in connection with Maxwell and Rahm's treatment of the theory of this method. These questions have to do with certain details rather than with the basic principle involved.

First, Eq. 4 implies that the energy dissipated as inelastic impact between the specimen and anvil is equal to the change in translational kinetic energy of the protruding portion of the specimen. That is

$$E_p = \frac{1}{2} M_A (v_1^2 - v_2^2)$$

where:

- $E_p$  = energy of plastic impact between sample and anvil,
- $M_A$  = mass of protruding sample,
- $v_1$  = linear velocity of sample prior to impact, and
- $v_2$  = linear velocity of sample after impact.

This equation is valid, however, only if the specimen is a freely moving body (except for its interaction with the anvil during impact), and if the only forces of interaction are those required to change the momentum of the specimen. Actually the specimen and rotor move as a single member until the instant of fracture is reached. The impulsive force during the initial part of the impact not only changes the linear momentum of the specimen, but also the angular momentum of the rotor. Thus, the force (and also  $E_p$ , the inelastic energy lost) is considerably larger than it would have been if the specimen had moved independently of the rotor during the entire impact. Therefore, the elimination of  $E_p$ , the inelastic energy of impact between specimen and

anvil, from the energy equation by means of the above relationship does not appear to be justified.

For convenience, we may represent  $E_p$  as the sum of two energies:

$$E_p = E_p' + E_p''$$

where:

- $E_p' = \frac{1}{2} M_A (v_1^2 - v_2^2)$  = plastic impact energy supplied by sample and
- $E_p''$  = plastic impact energy supplied by rotor.

It may then be demonstrated that, to a good approximation

$$E_b + E_p'' = \frac{1}{2} I (\omega_1^2 - \omega_2^2)$$

The right-hand side of this equation represents the quantity which is measured, that is, the change in kinetic energy of the rotor. This corresponds to  $E_b$ , the energy to break the sample, plus that portion of the plastic energy of impact supplied by the rotor,  $E_p''$ .

As Maxwell and Rahm indicate, it would seem undesirable to include any energy of plastic impact as part of the impact strength of the material. On the other hand, it is probable that the plastic impact energy involved is small relative to the energy to break the specimen, especially for the more brittle materials. Also, from the standpoint of a convenient, practical test I see no reason why the energy dissipated in plastic impact should not properly be included as part of the energy required to break the specimen in impact. If we are seeking a test method which correlates as closely as possible with service conditions, should we not include that energy which corresponds to the denting of an object when it

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<sup>2</sup> Earlham College, Richmond, Indiana.

receives a blow? An object consisting of ductile material owes part of its superior resistance to impact to its ability to deform (or dent) at the point of contact of the striking object. Ideally, of course, one might attempt to measure only the energy absorbed in deforming the uniform portion of the specimen. But it is difficult to visualize a simple impact test in which a soft or ductile sample does not absorb some energy at the point of contact of specimen and striking member. (Elaborate methods based on high-speed oscillographic stress-strain measurements can eliminate this energy, but then other difficulties arise.) Briefly then, for practical purposes, why not regard the energy dissipated between specimen and anvil (or hammer) as part of the impact resistance which characterizes a given material?

My second question has to do with Maxwell and Rahm's relation between "toss factor" of the Izod test, specific gravity of the sample, and "new method impact strength." My own attempts to deduce a relation between these quantities would lead me to think that an approximate relation of the following form exists:

$$\text{"Toss factor"} = \rho(a - bE)$$

where:

$\rho$  = density of sample,  
 $E$  = Izod impact strength, and  
 $a$  and  $b$  are constants.

Two adjustable constants are again available and it seems likely that the experimental data could be satisfactorily accounted for on this basis.

Regardless of the outcome of the questions raised above, it seems that the basic principle of operation of the "new method" has real possibilities for an improved "practical" impact test.

MESSRS. BRYCE MAXWELL AND L. F. RAHM (*authors' closure, by letter*).—The authors would like to thank Mr. Zinzow and Mr. Telfair for their comments and discussion.

Mr. Zinzow has brought up the question of the method of driving the rotor. In the machine described this was done with a laboratory stirring motor and a cone clutch. One rubber cone was attached to the shaft of the rotor and the other to the shaft of the motor. These two cones are engaged to bring the rotor up to speed and then disengaged so that the rotor is running free at the moment of impact.

Friction in the bearings and wind resistance can be allowed for if it is found to be a significant factor. In all the data presented here the friction was allowed for.

Mr. Telfair has raised a very interesting point. The authors have been concerned with the question of the distribution of the inelastic energy for some time and are indebted to Mr. Telfair for his remarks.

It is our belief that  $E_p'$  (plastic impact energy supplied by the specimen) is eliminated by this test but  $E_p''$  (plastic impact energy supplied by the rotor) is definitely included in the energy given by  $\frac{1}{2}I(\omega_1^2 - \omega_2^2)$ . As Mr. Telfair points out in a practical test of impact strength, the ease of plastic deformation (corresponding to  $E_p''$ ) has a direct bearing on the impact resistance of the material and should be included in the test. On the other hand,  $E_p'$  should not be included since it depends on factors similar to the toss factor.

The relationship proposed by Mr. Telfair between "toss factor" and density seems plausible. We would be interested to know whether it can be fitted to experimental data, and the constants  $a$  and  $b$  evaluated.

## The Preparation of Concrete for Airport Runways\*

By Inge Lyse<sup>1</sup>

### SYNOPSIS

This paper contains a brief description of the concrete and the method of construction used at the Sola Airfield, Norway. The runway slabs were only 8 in. thick and the flexural strength of the concrete was specified at 711 psi. (50 kg. per sq. cm.) at 28 days. Test loading on old concrete runways as well as new test slabs indicated that the 8-in. slabs should give sufficient load-carrying capacity. A simple vibrator paver was used for the construction of the runways. A special vibrator apparatus was used for controlling the dry consistency of the concrete. The question of special flexibility requirements for concrete for airport runways is discussed.

IN PLANNING the new concrete runways for Sola Airport, Norway, it seemed necessary to make a thorough investigation of the available aggregate sources located within reasonable distance of the airfield and to make test load studies of the old concrete runways as well as of new test

slabs. With the large areas covered by the new runways (about 320,000 sq. yd.) a small change in the thickness of the runways slab would be of considerable economic importance for the construction. As the Sola Airport is being placed in the A-1 class of international airports, the runways had to be constructed for the very largest type of airplanes.

The investigation of the concrete materials showed that concrete with a flexural strength of 711 psi. (50 kg. per sq. cm.) at 28 days could readily be obtained even with from 3 to 6 per

cent entrained air in the concrete. Six test slabs were made for the purpose of determining the load-carrying capacity of slabs of 4, 6, and 8-in. thickness, made of concrete having a flexural strength of 711 psi. at 28 days. The load tests of these slabs indicated that a thickness of 8 in. for the runways would be sufficient for the specified loading for the uniform and well-compacted subgrade at the Sola Airport. Load tests of the old runways indicated that the load-carrying capacity of the slabs increases with age due to an improved compacting of the subgrade and increase in the flexural strength of the concrete.

Scaling and disintegration of the concrete runways due to freezing and thawing are problems of importance in airport construction. It was deemed advisable to make as good provisions as possible for the prevention of such disintegration. The specification therefore called for an air-entrainment of from 3 to 6 per cent in the concrete.

The concrete would be placed by

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<sup>1</sup> Norway Institute of Technology, Trondheim, Norway.



means of a vibrating machine. The consistency of the concrete would therefore be very dry. A special vibrator apparatus was provided for controlling this dry consistency.

No reinforcement was used in the slabs or in the joints.

#### SPECIFICATIONS FOR THE CONCRETE

The material investigation had shown that natural sand deposits were located within a few miles of the airport and that coarse aggregate could be produced by crushing boulders found in abundance along the shore of the North Sea near the airport. But the contractor was free to choose any fine or coarse aggregate of accepted quality. The maximum size of the fine aggregate should not exceed  $\frac{3}{8}$  in. (10 mm.) and the maximum size of the coarse aggregate should not exceed  $1\frac{1}{2}$  in. (38 mm.). The coarse aggregate could contain up to 33 per cent gravel, the rest crushed rock. The cement should be ordinary Norwegian portland cement. Any accepted air-entraining agents were permitted and the contractor selected Darex AEA.

The concrete should have a flexural strength of at least 356 psi. (25 kg. per sq. cm.) at 7 days and 711 psi. (50 kg. per sq. cm.) at 28 days—as obtained by one-point loading on concrete beams of 4 by 5 by 20 in. (10 by 12.5 by 50 cm.) size. The concrete runways should be kept moist cured for at least 10 days.

#### METHOD OF CONSTRUCTION

The concrete was mixed at a central mixing plant located in one of the air-dromes of the airport. All materials were weighed for batching and the time of mixing was set at  $1\frac{1}{2}$  min. Because of the dry consistency of the concrete, the water content varied from 13.5 per cent to 14 per cent of the volume of the concrete. The cement content of the concrete varied from 500 to 550 lb. per cu. yd. (300 to 325 kg. per cu. m.). The concrete was transported from the mixer to the place of paving by means of ordinary dump trucks and spread in suitable thickness ahead of the paving machine by means of manpower. The paving machine was a Swedish paving vibrator manufactured by A. B. Vibroverken, Stockholm. It consists of a rigid frame on which is mounted a screed for leveling off the mass in front of the main vibrator; behind the screed is the main vibrator; behind this, two smaller vibrators placed somewhat diagonally, and finally the finishing screed. The concrete is thoroughly vibrated by the main vibrator. The diagonally placed vibrators smooth out the surface and the

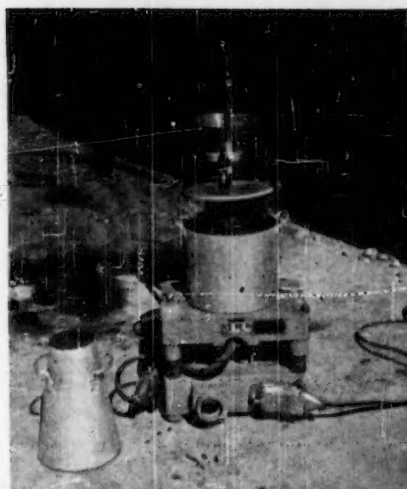


Fig. 1.—Vebe-meter for Measuring Consistency of Concrete.

final finishing screed gives the pavement its final shape.

#### MATERIALS

Rigid control of the materials for the concrete and of the concrete itself was maintained throughout the construction period. The sand was of excellent quality, but varied somewhat in gradation. All aggregate passing a  $\frac{3}{8}$ -in. sieve was classified as sand. The coarse aggregate was of one gradation

the top of the concrete and the table vibrator started. The time of vibration needed for the concrete to flow out and give a completely level surface in the container is measured very accurately. This time, given in seconds, is the Vebe-degree. A consistency of 10 deg. VB means a concrete which requires 10 sec. of vibration in the Vebe-meter for obtaining smooth surface of the concrete. This apparatus serves well for controlling the consistency of the very dry concrete used for vibration. But for concrete of plastic consistency the slump cone gives a better indication of the consistency. The consistency of the concrete was maintained at 10 to 12 deg. VB, corresponding to a slump of less than  $\frac{1}{2}$  in., as this gave the most satisfactory results for the paver.

The amount of entrained air was measured by the pressure method being recommended at this Annual Meeting by the Society's Committee C-9 for publication as tentative.<sup>2</sup> The air content varied from 3 per cent to 6 per cent with an average value of about 4.5 per cent for the construction completed to date. The contractor has recently agreed to limit the air content to between 3 and 5 per cent.

The strength of the concrete was controlled regularly by flexural tests of beams. The results for the first few weeks of construction are shown in

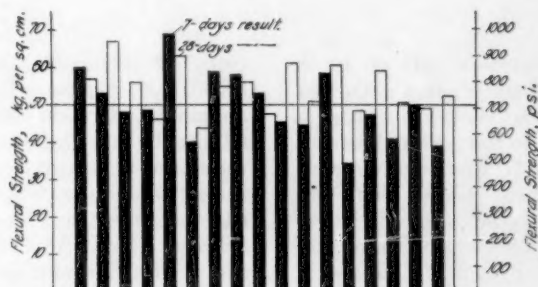


Fig. 2.—Flexural Strength of Concrete at Sola Airfield, Norway.

$\frac{3}{8}$  in. to  $1\frac{1}{2}$  in., and gave also a certain amount of variation in gradation. Some improvement in the uniformity of the concrete would no doubt have resulted from a further separation of the aggregates' sizes for the concrete. But the contractor's equipment was adapted to the two-sizes aggregates and this was approved for this job. The consistency of the concrete was controlled by means of a Vebe-meter (Fig. 1). This is an apparatus manufactured by the A. B. Vibroverken, Stockholm. It consists of a circular container placed on a table vibrator. A slump cone is set in this container and the concrete sample placed in the slump cone in the usual manner. The slump cone is removed and the slump measured. Then a glass plate is carefully placed on

Fig. 2 and give a fair indication of the variations obtained to date. It is noted that the 7-day flexural strengths are very high as compared to the 28-day results, and that individual results are as much as 10 per cent lower than the specified strength. These 10 per cent below requirements were permitted for individual results. The average flexural strength for all construction to date is 800 psi. (56.4 kg. per sq. cm.), well above the specification of 711 psi. (50 kg. per sq. cm.).

#### DISCUSSION

In considering the concrete for airport runways it should be kept in

<sup>2</sup> Tentative Method of Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 - 49 T), 1949 Book of A.S.T.M. Standards, Part 3.

mind that the flexibility as well as the flexural strength of the concrete slabs is of great importance for the load-carrying capacity of these slabs. The slabs transfer the load from the airplane to the subgrade, and a high flexibility of the slabs will more readily transfer the load to the subgrade than will a slab of small flexibility. Thus a thin

slab will give a relatively more effective load transfer than will a thick slab. Concrete for such runways should therefore be of special flexibility, but it would be difficult to make this a specification requirement. The problem of producing runway slabs with concrete of high flexural strength and high flexibility should receive further investigation. It might here be worth

while to consider the slab poured in two layers, with the top slab of, say, 4-in. thickness resting on a bottom slab which served as a semiflexible subgrade slab. The flexibility of the top slab would then be relatively high because of its small thickness and the bottom slab would serve well in distributing the load over the subgrade.

## The Use of Natural Anhydrite in Portland Cement\*

By W. C. Hansen<sup>1</sup> and J. O. Hunt<sup>1</sup>

### SYNOPSIS

Studies were made to determine the possibility of replacing gypsum wholly or in part by natural anhydrite in portland cements. Seven clinkers were ground with varying percentages of gypsum, and tests were made to determine the optimum amount of  $\text{SO}_3$  for each clinker. The clinkers were then ground with this optimum amount of  $\text{SO}_3$  in the form of mixtures of gypsum and natural anhydrite and the tests were repeated. A comparison of the results obtained with gypsum alone and with blends of gypsum and natural anhydrite showed that, with one clinker, 25 per cent and, with the others, 60 to 75 per cent of the gypsum could be replaced by natural anhydrite without adversely affecting the properties of the cements.

**C**ALCIUM sulfate occurs in nature as natural anhydrite,  $\text{CaSO}_4$ , and as gypsum,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ . The latter is used extensively in the portland cement industry to control the setting characteristics of the cement pastes. This use developed with the adoption of the rotary kiln and finer grinding of the clinker because these changes in the method of manufacture gave cements which, when mixed with water, set too rapidly for practical use. This type of fast setting, which is accompanied by a very rapid evolution of heat, came to be known as flash set.

The manner in which gypsum prevents flash set and controls the setting of cement pastes is not completely understood. In general, it is agreed that gypsum prevents the rapid set normally shown by tricalcium aluminate and that it causes the formation of cal-

cium sulfoaluminate,  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 31\text{H}_2\text{O}$ , in place of hydrated calcium aluminate. Certain investigators<sup>2</sup> attribute the retarding effect of gypsum on the setting of cement to the formation of the sulfoaluminate, which is assumed to have no cementing action, and others to a direct retardation of the rate of hydration of tricalcium aluminate. Probably, both of these actions are involved because the addition of gypsum to certain cements prevents the excessive heating of the paste which occurs when gypsum is omitted. This indicates a retardation of the rate of hydration. However, in the pastes containing gypsum, the gypsum is rather rapidly converted to sulfoaluminate which indicates that the rate at which the aluminates in the cement dissolve in water may not be retarded to a very great extent by gypsum.

Attempts<sup>2</sup> have been made to substitute natural anhydrite for gypsum, but none of these has led to the commercial use of anhydrite for this purpose. It is known that, in general, gypsum dissolves at a much more rapid rate than does natural anhydrite. Hence, if it is necessary for calcium sulfate to enter the solution rapidly to prevent flash set either through the retardation of the hydration of tricalcium aluminate or

through the formation of sulfoaluminate, it is conceivable that the rate of solution of natural anhydrite might be inadequate to supply the necessary concentration of  $\text{SO}_3$  in the liquid phase of the cement paste. However, the trend in the manufacture of portland cements has been to decrease the potential tricalcium aluminate contents of the cements and to quench the clinkers more thoroughly, which tends to leave the alumina in the clinker in the form of a glass, instead of permitting it to crystallize as aluminates. It is possible, therefore, that natural anhydrite might be substituted for gypsum in the cements of today without adverse effects.

The early investigators were concerned primarily with the effect of  $\text{SO}_3$  upon the setting times, although some recognized the effect upon the strengths. However, through the work of Lerch,<sup>3</sup> it is now known that the  $\text{SO}_3$  content of the cement affects the strength and volume constancy of concrete. Many present-day cements do not require gypsum to prevent flash set but do require it to develop high strengths at early ages and to give concretes which expand a minimum in a moist condition and contract a minimum in a dry condition. Lerch has shown that the amount of gypsum necessary to give these characteristics to the concrete varies with certain properties of the clinker. This has led to the concept that, for each portland-cement clinker, there is an amount of  $\text{SO}_3$  which, while preventing the cement paste from setting too rapidly, will yield the most satisfactory concrete from the standpoint of strength and volume change. That is, for each

**NOTE.**—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the author. Address all communications to A.S.T.M. Headquarters, 1916 Race St., Philadelphia 3, Pa.

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<sup>2</sup> See Lea and Desch, "The Chemistry of Cement and Concrete," Edward Arnold and Co., for a review of this subject.

<sup>3</sup> William Lerch, "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes," *Proceedings, Am. Soc. Testing Mats.*, Vol. 46, p. 1252 (1946).



clinker there is an optimum amount of gypsum which will give the most desirable properties to the concrete made from it.

Since natural anhydrite is an abundant natural resource for which no large-scale use has been found and since the compositions of cements have changed somewhat over the years and our knowledge of the function of  $\text{SO}_3$  in cement increased markedly with Lerch's work, it seemed desirable to reinvestigate the possibility of substituting natural anhydrite wholly or in part for gypsum in portland cement. Substitution of a part of the gypsum by natural anhydrite in cement would be a worth-while economic contribution because many gypsum mines contain veins of natural anhydrite which must be wasted at present. This paper presents the results of such a study conducted in the light of the work by Lerch.

#### EXPERIMENTAL PROCEDURE

Three types of portland-cement clinker (types I, II, and III of A.S.T.M. Specifications C 150)<sup>4</sup> were used in this investigation and the first step was to determine the optimum  $\text{SO}_3$  content for each. This was done by grinding each clinker in a laboratory mill at atmospheric temperatures with 0.5 and 3.5 per cent  $\text{SO}_3$  as gypsum and with 0.5 and 3.5 per cent  $\text{SO}_3$  as natural anhydrite. These grinds were blended to give cements with several different  $\text{SO}_3$  contents varying in steps of 0.25 per cent. Mortar cubes and 1 by 1 by 11½-in. prisms were prepared from these cements in accordance with A.S.T.M. Method of Test for Compressive Strength of Hydraulic Cement Mortars, (C 109-47).<sup>5</sup> The 2-in. cubes were tested for compressive strength at various ages, and the prisms were tested for volume change when stored in water and in air (Tables I to VII, inclusive). The prisms were stored in the molds in the moist cabinet at 70 F. for 24 hr. and then were removed from the molds and stored in water at 70 F. Three prisms of each kind were stored continuously in water and three of each kind were removed from the water at the age of 7 days and stored in the laboratory air, and the changes in the lengths of the prisms were measured periodically. The tests with the prisms were not made with the cements containing natural anhydrite.

After the optimum  $\text{SO}_3$  content for each cement was established, blends of the cements were made to give four or five cements of that  $\text{SO}_3$  content in

TABLE I.—RESULTS OF PHYSICAL TESTS FOR CEMENTS OF CLINKER A.

SO <sub>2</sub> Content, per cent	Normal Con- sistency	Setting Time Gulmore		Compressive Strength, psi., at Age Indicated <sup>a</sup>			Length Change of Mortar Prisms at 7 Days, per cent	
		Initial	Final	1 day	3 day	7 day	Expansion in Water <sup>b</sup>	Contraction in Air <sup>b</sup>
SO <sub>2</sub> FROM Gypsum								
1.50.....	24.5	4:00	7:15	484	1449	2900	...	...
1.75.....	24.5	4:30	7:20	708	1640	2717	...	...
2.00.....	23.0	4:40	7:25	752	1882	2875	0.006	0.062
2.25.....	24.0	4:35	7:10	698	1970	2938	0.006	0.061
2.50.....	23.5	4:05	7:15	665	1874	2867	0.005	0.054
2.75.....	23.0	5:05	7:55	649	1843	3017	0.004	0.054
3.00.....	23.5	4:05	7:05	602	1586	2692	0.022	0.065
SO <sub>2</sub> FROM ANHYDRITE								
1.75.....	26.0	2:40	6:40	585	1527	2471	...	...
2.00.....	23.5	2:10	6:30	663	1606	2542	...	...
2.25.....	23.0	2:45	7:00	632	1630	2542	...	...
2.50.....	24.0	3:40	6:55	614	1757	2700	...	...
2.75.....	24.0	4:10	7:35	572	1789	2717	...	...
3.00.....	24.0	3:35	7:00	559	1686	2692	...	...
SO <sub>2</sub> FROM MIXTURES OF ANHYDRITE PLUS GYPSUM								
2.00 (a) <sup>c</sup> ...	24.0	4:00	6:50	651	1729	2784	0.005	0.059
2.00 (b)....	24.5	3:50	6:45	721	1760	2834	0.006	0.063
2.00 (c)....	23.5	4:00	6:50	743	1754	2883	0.008	0.060
2.00 (d)....	24.0	3:50	6:40	723	1694	2700	0.007	0.061

<sup>a</sup> A.S.T.M. Method C 109-47.

<sup>b</sup> Expansion during 6 days in water after 1 day in mold; contraction during 7 days in laboratory air after 1 day in mold and 6 days in water.

<sup>c</sup> (a) = 25.0 per cent anhydrite; (b) = 37.5 per cent anhydrite; (c) = 50.0 per cent anhydrite; and (d) = 62.5 per cent anhydrite.

TABLE IA.— $\text{SO}_3$  CONTENT OF EXTRACTS FROM MORTAR SPECIMENS AT AGE INDICATED, G. PER LITER.

$\text{SO}_3$ Content, per cent	Gypsum		Anhydrite		Mixtures of Anhydrite and Gypsum Containing 2.0 per cent $\text{SO}_3$ with per cent Anhydrite Indicated					
					37.5		50.0		62.5	
	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.
1.75..	0.01	0.01	0.20	0.03	0.15	0.01	0.17	0.02	0.18	0.02
2.00..	0.08	0.01	0.35	0.13	...	...	...	...	...	...
2.25..	0.33	0.08	0.43	0.25	...	...	...	...	...	...
2.50..	0.77	0.36	0.57	0.44	...	...	...	...	...	...
2.75..	0.75	0.71	0.68	0.57	...	...	...	...	...	...
3.00..	1.32	1.17	0.68	0.57	...	...	...	...	...	...

which from 25.0 to 77.5 per cent of the  $\text{SO}_3$  was in the form of natural anhydrite. The strengths and volume change characteristics of these cements were then determined by the procedures outlined above.

Mr. Lerch suggested that it might be possible to use the data obtained from extracts of mortar prisms to determine whether or not the cement contained the optimum amount of gypsum. The procedure being studied presently by the Subcommittee on  $\text{SO}_3$  Content, of A.S.T.M. Committee C-1 on Cement is to make two mortar specimens (A.S.T.M. Specifications C 109 mortars), cure them in the moist cabinet, and make extracts at the ages of 18 and 24 hr. In this procedure, the specimen is crushed to pass a No. 10 sieve, 200 g. of the powdered specimen is stirred with 50 ml. of distilled water for 10 min., the slurry is filtered and the filtrate is analyzed for its  $\text{SO}_3$  content. This procedure was followed in this investigation.

To demonstrate whether or not the mixtures of anhydrite and gypsum were

equal to gypsum alone in maintaining a high concentration of  $\text{SO}_3$  in the liquid phase of the paste of cement and water, an extraction test was made on certain cements. In this test, pastes of 100 g. of cement with 75 ml. of distilled water were shaken continuously at room temperature for several periods of time, and the filtrates were analyzed for  $\text{SO}_3$ .

The normal consistencies and setting times of the cements used in this investigation were determined by the standard A.S.T.M. procedures, and the heats of hydration of six of the cements were determined in a conduction calorimeter by the procedure outlined by Lerch.<sup>3</sup> The results of the various tests are given in Tables I to VIII.

The gypsum used in this study was a high-grade commercial rock gypsum, and the natural anhydrite was a carefully selected natural product. The compositions of these were as follows:

<sup>4</sup> Standard Specifications for Portland Cement (C 150-47), 1947 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

<sup>5</sup> 1947 Supplement to Book of A.S.T.M. Standards, Part II, p. 43.

	Composition, per cent by weight							Loss at	
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Moisture at 45 C.	230 C. <sup>a</sup>	950 C. <sup>b</sup>
Gypsum....	2.50	1.05	0.42	31.40	0.86	42.34	0.01	18.37	2.76
Anhydrite..	0.00	0.03	0.02	40.86	0.20	57.62	0.01	0.75	0.52

<sup>a</sup> Loss at 230 C. after drying at 45 C.

<sup>b</sup> Loss at 950 C. after loss at 230 C.

The compositions of the clinkers used in the study were as follows:

Clinker	Composition, per cent by weight								Loss on ignition, per cent.
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O
TYPE I									
A.....	22.06	5.96	2.62	63.76	2.52	0.06	0.15	0.61	0.17
B.....	21.66	5.24	3.26	63.87	3.38	0.08	0.38	0.49	0.21
C.....	22.18	6.73	2.71	65.60	1.10	0.15	0.10	0.49	0.19
TYPE II									
D.....	21.76	4.73	3.59	64.61	2.50	0.07	0.13	0.50	0.12
E.....	23.14	5.16	3.96	65.08	1.12	0.16	0.16	0.45	0.20
TYPE III									
F.....	21.38	4.83	3.03	64.43	3.16	0.10	0.17	0.41	0.19
G.....	21.54	6.76	2.60	66.22	1.10	0.16	0.09	0.47	0.17

## DISCUSSION OF RESULTS OF PHYSICAL TESTS

At present, there is no established criterion for selecting the optimum amount of SO<sub>3</sub> for a given clinker, but it appears that the optimum is the amount which will be almost completely combined with the cement in 24 hr. when mortars are stored in a moist atmosphere at approximately 70 F. In this study, a combination of two criteria was used. The first was the compressive strengths at 1 day. The second was the SO<sub>3</sub> content of the extracts from the mortar specimens. If the quantity of SO<sub>3</sub> as gypsum which gave the highest strength at 1 day did not give more than 0.05 g. per liter of SO<sub>3</sub> in the extract from the mortar specimen at the age of 24 hr., this amount of SO<sub>3</sub> was selected as the optimum. If this quantity of SO<sub>3</sub> gave a concentration of SO<sub>3</sub> in the extract greater than 0.05 g. per liter, the greatest quantity of SO<sub>3</sub> which gave less than 0.05 g. per liter of SO<sub>3</sub> in the extract from the mortar specimen was selected as the optimum.

As pointed out under the section on Experimental Procedure, each clinker was first studied with various percentages of SO<sub>3</sub> in the form of gypsum. Then the optimum SO<sub>3</sub> content was selected from the data for the strength and extraction tests and cements were prepared with this SO<sub>3</sub> content obtained from mixtures of gypsum and natural anhydrite. For example, the data in Tables I and IA show that clinker A gave the highest strength at 1 day when the SO<sub>3</sub> content as gypsum was 2.0 per cent and that the extract from the mortar of this cement at the age of 24 hr. did not contain more than 0.05 g. per liter

of SO<sub>3</sub>. Hence, 2.0 per cent SO<sub>3</sub> was selected as the optimum.

### Type I Cements:

The data for type I cements prepared from clinker from three different plants are given in Tables I to III and IA to IIIA, inclusive. The optimum SO<sub>3</sub> content for clinker A was selected as 2.0 per cent as explained above. It may be

seen, Table I, that when the 2.0 per cent SO<sub>3</sub> was obtained entirely from anhydrite the setting times and strengths were appreciably lower than when the 2.0 per cent of SO<sub>3</sub> was derived from gypsum. However, when the 2.0 per cent of SO<sub>3</sub> was obtained from mixtures of gypsum and anhydrite in which the anhydrite content ranged from 25.0 to 62.5 per cent, the setting times and strengths were very similar to those of the cement containing 2.0 per cent SO<sub>3</sub> as gypsum. Likewise, the results of the expansion, contraction, and extraction tests of the mortar from the cements made with the blends of gypsum and anhydrite agreed very closely with those of the specimens made with the cement having 2.0 per cent SO<sub>3</sub> as gypsum. Hence, it appears that mixtures containing up to 62.5 per cent natural anhydrite could be used to replace gypsum with clinker A without adversely affecting the properties of the concrete made from the cement.

The results for clinker B are given in Table II and IIA. Here again, 2.0 per cent was selected as the optimum SO<sub>3</sub> content on the basis of the strengths at 1 day and the SO<sub>3</sub> contents of the extracts from mortar specimens. This clinker differs from clinker A in that complete substitution of anhydrite for gypsum had little if any effect upon the

TABLE II.—RESULTS OF PHYSICAL TESTS FOR CEMENTS OF CLINKER B.

SO <sub>2</sub> Content, per cent	Normal Con- sistency	Setting Time Gillmore		Compressive Strength, psi., at Age Indicated <sup>a</sup>			Length Change of Mortar Prisms at 7 days, per cent	
		Initial	Final	1 day	3 day	7 day	Expansion in water <sup>b</sup>	Contract- ion in air <sup>b</sup>
SO <sub>2</sub> FROM GYPSUM								
2.00.....	25.0	5:40	8:20	669	1823	2758	0.005	0.057
2.25.....	25.0	5:30	8:30	661	1900	2704	0.003	0.053
2.50.....	25.0	5:20	8:30	576	1897	2717	0.005	0.054
2.75.....	25.0	5:30	8:40	664	2042	2983	0.004	0.054
SO <sub>2</sub> FROM ANHYDRITE								
2.00.....	25.5	5:15	8:40	615	1808	2700	...	...
2.25.....	25.5	5:20	8:45	620	1833	2650	...	...
2.50.....	25.5	5:25	8:40	731	1944	2800	...	...
2.75.....	25.5	5:20	8:45	735	1984	2850	...	...
SO <sub>2</sub> FROM MIXTURES OF ANHYDRITE PLUS GYPSUM								
2.00 (a) <sup>c</sup> ...	25.0	5:20	7:15	803	1818	2742	0.008	0.033
2.00 (b)....	25.0	5:20	8:15	718	1850	2667	0.009	0.032
2.00 (c)....	25.0	5:25	7:15	707	1937	2692	0.009	0.033
2.00 (d)....	25.0	5:30	8:25	750	1804	2525	0.008	0.037

<sup>a</sup> A.S.T.M. Method C 109 - 47 mortar.

<sup>b</sup> Expansion during 6 days in water after 1 day in mold; contraction during 7 days in laboratory air after 1 day in mold and 6 days in water.

(a) = 25.0 per cent anhydrite; (b) = 37.5 per cent anhydrite; (c) = 50.0 per cent anhydrite; and (d) = 62.5 per cent anhydrite.

TABLE IIA.—SO<sub>3</sub> CONTENT OF EXTRACTS FROM MORTAR SPECIMENS AT AGE INDICATED, G. PER LITER.

SO <sub>3</sub> Content, per cent	Gypsum		Anhydrite		Mixtures of Anhydrite and Gypsum Containing 2.0 per cent SO <sub>3</sub> with per cent Anhydrite Indicated					
					37.5		50.0		62.5	
	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.
2.00..	0.17	0.03	0.49	0.13	0.11	0.11	0.11	0.02	0.13	0.02
2.25..	0.51	0.13	0.71	0.33	...	...	...	...	...	...
2.50..	0.90	0.36	0.92	0.68	...	...	...	...	...	...
2.75..	1.58	0.67	1.04	0.89	...	...	...	...	...	...



TABLE III.—RESULTS OF PHYSICAL TESTS FOR CEMENTS OF CLINKER C.

SO <sub>2</sub> Content, per cent	Normal Consistency	Setting Time Gillmore		Compressive Strength, psi., at Age Indicated <sup>a</sup>			Length Change of Mortar Prisms at 7 Days, per cent	
		Initial	Final	1 day	3 day	7 day	Expansion in Water <sup>b</sup>	Contraction in air <sup>b</sup>
SO <sub>2</sub> FROM GYPSUM								
1.50.....	25.0	4:00	7:35	360	1488	2900	0.012	0.089
1.75.....	25.0	4:30	7:55	453	1583	2933	0.011	0.080
2.00.....	24.5	4:25	7:40	527	1605	2933	0.010	0.067
2.25.....	24.5	4:20	7:35	642	1725	3175	0.007	0.071
2.50.....	24.5	4:25	7:20	623	1702	3125	0.006	0.056
2.75.....	24.5	4:30	7:20	581	1720	2825	0.008	0.055
3.00.....	24.5	4:20	7:25	557	1777	2975	0.006	0.058
SO <sub>2</sub> FROM ANHYDRITE								
1.50.....	31.0	3:50	8:35	377	1593	2967	...	...
1.75.....	31.0	3:05	7:15	409	1530	2900	...	...
2.00.....	31.0	4:00	8:35	406	1496	2792	...	...
2.25.....	30.0	3:55	8:30	533	1717	2808	...	...
2.50.....	29.0	3:55	7:50	591	1753	2838	...	...
2.75.....	29.0	3:50	7:55	661	1705	2892	...	...
3.00.....	29.0	3:50	8:10	715	1804	2825	...	...
SO <sub>2</sub> FROM MIXTURES OF ANHYDRITE PLUS GYPSUM								
2.75 (a) <sup>c</sup> ...	27.5	4:55	7:30	666	1857	3033	0.010	0.041
2.75 (b) <sup>c</sup> ...	28.0	4:40	7:25	689	1838	2975	0.008	0.031
2.75 (c) <sup>c</sup> ...	28.0	4:40	7:40	667	1831	2992	0.013	0.039
2.75 (d) <sup>c</sup> ...	28.0	4:45	7:35	599	1773	3092	0.011	Broken

<sup>a</sup> A.S.T.M. Method C 109-47 mortar.<sup>b</sup> Expansion during 6 days in water after 1 day in mold; contraction during 7 days in laboratory air after 1 day in mold and 6 days in water.<sup>c</sup> (a) = 25.0 per cent anhydrite; (b) = 37.5 per cent anhydrite; (c) = 50.0 per cent anhydrite; and (d) = 62.5 per cent anhydrite.TABLE IIIA.—SO<sub>2</sub> CONTENT OF EXTRACTS FROM MORTAR SPECIMENS AT AGE INDICATED, G. PER LITER.

SO <sub>2</sub> Content, per cent	SO <sub>2</sub> FROM SOURCE INDICATED									
	Gypsum		Anhydrite		Mixtures of Anhydrite and Gypsum Containing 2.75 per cent SO <sub>2</sub> with per cent of Anhydrite Indicated					
					37.5		50.0		62.5	
	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.
2.00..	0	0	0.01	0	...	...	...	...	...	...
2.25..	0	0	0.01	0	...	...	...	...	...	...
2.50..	0	0	0.04	0	...	...	...	...	...	...
2.75..	0	0	0.13	0	0.12	0	0.11	0	0.01	0

setting times and strengths. The expansions of the mortars were slightly greater and the contractions were somewhat smaller for the cements made with blends of gypsum and anhydrite than for those made with all gypsum. However, the differences are small and, since the specimens were made at different times, they probably are not significant.<sup>6</sup> In this case, it appears that gypsum could be replaced completely by natural anhydrite.

The data for clinker C are given in Tables III and IIIA. With this clinker, the maximum strength at 1 day was obtained with 2.25 per cent SO<sub>2</sub>, but the extracts from the mortars contained no SO<sub>2</sub> with as much as 2.75 per cent SO<sub>2</sub> in the cement. Since increasing the SO<sub>2</sub> content from 2.25 to 2.75 per cent caused very little change in the strength at 1 day, 2.75 per cent was selected as

<sup>6</sup> The prisms for the contraction studies were stored in a room in which the temperature and relative humidity were not controlled. Accordingly, differences in the contractions between the two sets of specimens may be due to differences in the storage conditions.

<sup>7</sup> This calorimeter is similar to the one described by Lerch.

the quantity to use in the blends. Substitution of anhydrite for gypsum with this clinker increased the amount of water required for normal consistency. However, from the standpoint of the properties such as setting times, strength, expansion, and contraction, replacement of gypsum by anhydrite with this clinker does not appear to be harmful.

Because of this indication of fast setting with this clinker, cements were made containing 2.25 per cent SO<sub>2</sub> in which the 100 per cent, 62.5 per cent and 37.5 per cent of the SO<sub>2</sub> was present as gypsum. The heats of hydration of these cements were determined at 70 F in a conduction calorimeter,<sup>7</sup> and the results are given in Table VIII. The differences between the amounts of heat liberated at any period are not very great for the three cements, but the two cements containing the natural anhydrite tended to liberate heat at slightly greater rates during the early periods than did the one with gypsum alone. These data, therefore, confirm to some extent those for normal consistency which indicates that, although anhydrite and mixtures of anhydrite and gypsum prevent flash set in cements from clinker C, the rate of hydration is more effectively retarded during the early periods by gypsum than by mixtures of gypsum and anhydrite.

#### Type II Cements:

Tables IV and IVA give the results for clinker D. With this clinker, 2.0 per cent SO<sub>2</sub> gave the highest strength at 1

TABLE IV.—RESULTS OF PHYSICAL TESTS FOR CEMENTS OF CLINKER D.

SO <sub>2</sub> Content, per cent	Normal Con- sistency	Setting Time Gillmore		Compressive Strength, psi., at Age Indicated <sup>a</sup>			Length Change of Mortar Prisms at 7 Days, per cent	
		Initial	Final	1 day	3 day	7 day	Expansion in Water <sup>b</sup>	Contraction in air <sup>b</sup>
SO <sub>2</sub> FROM GYPSUM								
1.50.....	23.5	4:05	6:55	588	1514	2392	0.012	0.049
1.75.....	23.5	3:50	6:50	631	1664	2583	0.011	0.047
2.00.....	23.5	3:45	7:05	663	1825	2783	0.010	0.050
2.25.....	23.5	4.15	7.10	533	1748	2850	0.006	0.055
2.50.....	23.5	4:40	8:00	568	1712	2550	0.010	0.054
SO <sub>2</sub> FROM ANHYDRITE								
1.50.....	25.5	1:30	6:10	537	1544	2583	...	...
1.75.....	26.0	3:20	7:10	555	1626	2600	...	...
2.00.....	26.0	3:25	7:20	604	1767	2767	...	...
2.25.....	26.0	4:20	7:25	590	1825	2858	...	...
2.50.....	26.0	4:15	7:40	598	1891	2892	...	...
SO <sub>2</sub> FROM MIXTURES OF ANHYDRITE PLUS GYPSUM								
1.75 (a) <sup>c</sup> ...	23.0	4:00	7:00	690	1912	2825	0.008	0.048
1.75 (b)....	23.0	4:10	6:55	661	1797	2683	0.008	0.045
1.75 (c)....	23.0	4:05	6:55	700	1673	2533	0.004	0.046
1.75 (d)....	23.5	4:00	7:00	708	1742	2692	0.008	0.049
1.75 (e)....	23.5	4:10	6:55	578	1605	2458	0.008	0.050

<sup>a</sup> A.S.T.M. Method C 109-47 mortar.<sup>b</sup> Expansion during 6 days in water after 1 day in mold; contraction during 7 days in laboratory air after 1 day in mold and 6 days in water.<sup>c</sup> (a) = 28.5 per cent anhydrite; (b) = 42.3 per cent anhydrite; (c) = 50.0 per cent anhydrite; (d) = 57.2 per cent anhydrite; and (e) = 71.5 per cent anhydrite.

TABLE IVA.—SO<sub>3</sub> CONTENT OF EXTRACTS FROM MORTAR SPECIMENS AT AGE INDICATED, G. PER LITER.  
SO<sub>3</sub> FROM SOURCE INDICATED

SO <sub>3</sub> Content, per cent	Gypsum		Anhydrite		Mixtures of Anhydrite and Gypsum Containing 1.75 per cent SO <sub>3</sub> with per cent of Anhydrite Indicated					
	18 hr.	24 hr.	18 hr.	24 hr.	50.0		57.2		71.5	
					18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.
1.50..	0.03	0.01	0.02	0.01	...	...	...	...	...	...
1.75..	0.08	0.02	0.10	0.01	0.06	0	0.09	0	0.07	0
2.00..	0.27	0.10	0.23	0.05	...	...	...	...	...	...
2.25..	0.58	0.24	0.41	0.18	...	...	...	...	...	...
2.50..	1.23	0.62	0.58	0.39	...	...	...	...	...	...

day but the concentration of SO<sub>3</sub> in the extract from the mortar at the age of 24 hr. exceeded 0.05 g. per liter. Hence, 1.75 per cent SO<sub>3</sub> was selected as the optimum. Complete replacement of gypsum by anhydrite increased the amount of water required for normal consistency, which indicates that the anhydrite is not as effective as gypsum in retarding the initial stiffening. However, mixtures of gypsum and anhydrite with anhydrite up to 71.5 per cent gave cements which, in all respects, were comparable to those made with gypsum alone.

With clinker E, Table V, 2.25 per cent SO<sub>3</sub> gave the highest strength at 1 day and the extract from the mortar specimen at the age of 24 hr. contained less than 0.05 g. per liter of SO<sub>3</sub>. Accordingly, 2.25 per cent SO<sub>3</sub> was selected as the optimum. Here again, the normal consistencies were high when all anhydrite was used. The inability of all anhydrite to control the initial setting is demonstrated in Table V where 1.50 per cent SO<sub>3</sub> as gypsum gave normal setting times, but 1.50 per cent SO<sub>3</sub> as anhydrite did not. The cements made with the mixtures of gypsum and anhydrite with anhydrite up to 77.5 per cent tended to have slightly shorter setting times and slightly lower strengths than did the cement with 2.25 per cent SO<sub>3</sub> as gypsum. However, the cements with the mixtures of anhydrite and gypsum were tested several weeks after the other cements and some of the difference might be due to aging of the cement, because only small quantities of cement were prepared. In general, therefore, it appears that mixtures of anhydrite and gypsum may be used with the two type II cement clinkers.

#### Type III Cements:

The results for two type III cements are given in Table VI, VII, and VIIA. With clinker F, Tables VI and VIIA, next to the highest strength at 1 day was obtained with 3.00 per cent SO<sub>3</sub> as gypsum and the extract from the mortar of this cement at the age of 1 day contained less than 0.05 g. per liter of

mortar prisms were not very excessive when 25 per cent of the SO<sub>3</sub> was obtained from anhydrite. Hence, it appears that gypsum containing up to 25 per cent anhydrite could be used with this clinker but that greater amounts of anhydrite might be harmful. No explanation can be given at this time as to why the strengths decreased so much as the proportion of anhydrite to gypsum increased when blends of anhydrite and gypsum were used in place of all gypsum, inasmuch as complete substitution

TABLE V.—RESULTS OF PHYSICAL TESTS FOR CEMENTS OF CLINKER E.

SO <sub>3</sub> Content, per cent	Normal Con- sistency	Setting Time Gillmore		Compressive Strength, psi., at Age Indicated <sup>a</sup>			Length Change of Mortar Prisms at 7 Days, per cent	
		Initial	Final	1 day	3 day	7 day	Expansion in Water <sup>b</sup>	Contraction in Air <sup>b</sup>
SO <sub>2</sub> FROM GYPSUM								
1.50.....	23.5	5:40	8:05	625	1794	3025	0.004	0.061
1.75.....	23.5	5:35	8:00	708	1898	3083	0.003	0.055
2.00.....	23.5	5:25	7:55	795	1983	3129	0.004	0.052
2.25.....	23.5	5:20	7:40	840	2082	3084	0.006	0.051
2.50.....	23.5	5:20	8:05	791	2044	3192	0.103	0.044
SO <sub>2</sub> FROM ANHYDRITE								
1.50.....	25.0	0:10	6:35	526	1742	3033	...	...
1.75.....	25.0	2:50	7:05	527	1720	2950	...	...
2.00.....	25.0	3:30	7:00	531	1816	2821	...	...
2.25.....	26.0	4:10	6:15	596	1854	2954	...	...
2.50.....	25.0	3:50	7:05	665	2014	3046	...	...
SO <sub>2</sub> FROM MIXTURES OF ANHYDRITE PLUS GYPSUM								
2.25 (a) <sup>c</sup> ...	23.0	4:20	7:10	700	1838	2758	0.009	0.052
2.25 (b)...	23.0	4:10	7:10	675	1827	2750	0.010	0.049
2.25 (c)...	23.5	4:10	7:15	729	1867	2858	0.012	0.049
2.25 (d)...	23.5	4:20	7:15	648	1840	2750	0.011	0.048
2.25 (e)...	23.5	4:10	7:05	643	1922	2875	0.010	0.047

<sup>a</sup> A.S.T.M. Method C 109 - 47 mortar.  
<sup>b</sup> Expansion during 6 days in water after 1 day in mold; contraction during 7 days in laboratory air after 1 day in mold and 6 days in water.  
<sup>c</sup> (a) = 32.5 per cent anhydrite; (b) = 44.5 per cent anhydrite; (c) = 50.0 per cent anhydrite; (d) = 66.5 per cent anhydrite; and (e) = 77.5 per cent anhydrite.

TABLE VA.—SO<sub>3</sub> CONTENT OF EXTRACTS FROM MORTAR SPECIMENS AT AGE INDICATED, G. PER LITER.

SO <sub>3</sub> Content, per cent	Gypsum		Anhydrite		Mixtures of Anhydrite and Gypsum Containing 2.25 per cent SO <sub>3</sub> with per cent of Anhydrite Indicated					
	18 hr.	24 hr.	18 hr.	24 hr.	50.0		66.5		77.5	
					18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.
1.50..	0.01	0.01	0.01	0.01	...	...	...	...	...	...
1.75..	0.01	0	0.04	0.00	...	...	...	...	...	...
2.00..	0.10	0	0.17	0.02	...	...	...	...	...	...
2.25..	0.34	0.04	0.28	0.08	0.05	0	0.01	0	0.05	0
2.50..	0.74	0.17	0.38	0.17	...	...	...	...	...	...

SO<sub>3</sub>. This was, therefore, selected as the optimum SO<sub>3</sub> content. The setting times were shortened by approximately an hour when anhydrite was completely substituted for gypsum and the strengths were decreased slightly. There was a tendency for the setting times to decrease as more and more anhydrite was used in the mixtures of anhydrite and gypsum and the strengths markedly decreased as the quantities of anhydrite increased. Also, the expansions of the mortar prisms from the cements made with the blends of anhydrite and gypsum were considerably greater than were those of the prisms from the cement containing all gypsum. The loss in strength and the increase in the expansions of the

of anhydrite for gypsum produced only slight decrease in the strengths.

Clinker G, Table VII, gave the highest strengths at 1 day with 2.75 per cent SO<sub>3</sub> as gypsum, but the strengths did not change significantly with SO<sub>3</sub> contents up to 3.50 per cent. Also, it was not until 3.50 per cent SO<sub>3</sub> was used that the concentration of SO<sub>3</sub> in the extract from the mortar at the age of 24 hr. exceeded 0.05 g. per liter. Hence, 3.25 per cent SO<sub>3</sub> was selected as the optimum. Complete substitution of anhydrite for gypsum increased the normal consistencies, decreased the setting times and strengths. Replacement of gypsum by anhydrite up to 62.5 per cent anhydrite had no significant effect upon the nor-



TABLE VI.—RESULTS OF PHYSICAL TESTS FOR CEMENTS OF CLINKER F.

SO <sub>2</sub> Content, per cent	Normal Consistency	Setting Time Gillmore		Compressive Strength, psi., at Age Indicated <sup>a</sup>			Length Change of Mortar Prisms at 7 Days, per cent	
		Initial	Final	1 day	3 day	7 day	Expansion in Water <sup>b</sup>	Contraction in Air <sup>b</sup>
SO <sub>2</sub> FROM GYPSUM								
2.50.....	25.0	3:15	5:30	1818	3896	5283	0.608	0.073
2.75.....	25.0	3:20	5:20	1907	3675	5108	0.004	0.069
3.00.....	25.0	3:10	5:20	1935	3850	5096	0.006	0.066
3.25.....	25.0	3:10	5:15	1862	3808	4984	0.009	0.063
3.50.....	25.0	3:15	5:15	1964	3833	5025	0.003	0.066
SO <sub>2</sub> FROM ANHYDRITE								
2.50.....	25.5	2:10	4:50	1789	3350	4409	...	...
2.75.....	25.0	2:15	4:50	1698	3308	4300	...	...
3.00.....	25.0	2:10	4:45	1692	3383	4384	...	...
3.25.....	25.0	2:15	4:45	1603	3383	4459	...	...
3.50.....	25.0	2:10	5:00	1789	3567	4538	...	...
SO <sub>2</sub> FROM MIXTURES OF ANHYDRITE PLUS GYPSUM								
3.00 (a) <sup>c</sup> ...	25.0	3:50	5:50	1875	3475	4475	0.011	0.040
3.00 (b).....	25.0	3:50	5:45	1858	3267	4467	0.016	0.041
3.00 (c).....	25.0	3:45	5:45	1694	2975	4417	0.016	0.040
3.00 (d).....	25.0	3:25	5:40	1459	2683	4167	0.015	Broken
3.00 (e).....	25.0	3:15	5:30	1838	2208	3825	0.014	0.037

<sup>a</sup> A.S.T.M. Method C 109-47 mortar.<sup>b</sup> Expansion during 6 days in water after 1 day in mold; contraction during 7 days in laboratory air after 1 day in mold and 6 days in water.<sup>c</sup> (a) = 25.0 per cent anhydrite; (b) = 41.5 per cent anhydrite; (c) = 50.0 per cent anhydrite; (d) = 58.5 per cent anhydrite; and (e) = 75.0 per cent anhydrite.TABLE VII.—SO<sub>2</sub> CONTENT OF EXTRACTS FROM MORTAR SPECIMENS AT AGE INDICATED, G. PER LITER.

SO <sub>2</sub> Content, per cent	Gypsum		Anhydrite		Mixtures of Anhydrite and Gypsum Containing 3.00 per cent SO <sub>2</sub> with per cent of Anhydrite Indicated					
					50.0		58.5		75.0	
	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.
2.50.....	0.01	0.01	0.08	0.02	...	...	...	...	...	...
2.75.....	0.14	0.05	0.15	0.03	...	...	...	...	...	...
3.00.....	0.12	0.03	0.29	0.09	0.54	0.27	0.27	0.18	0.59	0.20
3.25.....	0.59	0.36	0.62	0.31	...	...	...	...	...	...
3.50.....	0.87	0.47	0.83	0.46	...	...	...	...	...	...

mal consistencies, but it slightly decreased the initial setting times and increased the strengths. Hence, it appears that anhydrite up to at least 62.5 per cent may be used to replace gypsum with this clinker.

The heats of hydration were determined in the conduction calorimeter for cements made with clinker F containing 3.25 per cent SO<sub>2</sub> in which 100 per cent, 62.5 per cent, and 37.5 per cent of the SO<sub>2</sub> was present as gypsum. The results are given in Table VIII, from which it may be seen that the rate at which heat was liberated during the early periods increased very slightly as the gypsum content decreased. It appears, therefore, that the effectiveness of the mixture of gypsum and anhydrite in retarding the rate of reaction in the early periods decreases as the gypsum content decreases.

The results of this study with seven clinkers of three types show that large substitutions of anhydrite for gypsum may be made with six of them without adversely affecting the properties of the concrete produced from the cements. They also indicate that the seventh clinker could be used with gypsum containing up to about 25 per cent anhydrite

without seriously affecting the properties of the concrete.

## GENERAL DISCUSSION

It was pointed out in the introduction

that the mechanism by which gypsum controls the setting of portland cement is not fully understood. However, it is known that certain clinkers will produce cement which will flash set unless mixed with certain minimum amounts of calcium sulfate, and it is also known that calcium sulfate accelerates the rate of reaction of the cement with water. Hence, in some cements, gypsum performs the dual function of preventing flash set and accelerating the rate of reaction; whereas, in other, the latter is its only function.

It has been believed generally that natural anhydrite could not be used to control the rate of setting and hardening of portland cement because its rate of solution was not sufficiently rapid. In this connection, it is to be remembered that, when cement clinker is ground with either gypsum or natural anhydrite, the particles of the resulting powder vary in size over the range of approximately 1 to 75  $\mu$ . These particles, being fragments of broken crystals, tend to dissolve at a greater rate than do perfect crystals of the same sizes, and the finer particles dissolve more rapidly than do the coarser particles.

Studies by Kalousek, Jumper, and Tregoning<sup>8</sup> and by others have shown that the principal components of the liquid phase of a cement paste at early ages are CaO, K<sub>2</sub>O, Na<sub>2</sub>O, and SO<sub>2</sub> and that SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and, in some cases, Cr<sub>2</sub>O<sub>3</sub> are present in very minor amounts. Hence for practical purposes, the liquid phase of a cement paste may be considered as a solution of calcium and

<sup>8</sup> G. L. Kolousek, C. H. Jumper, and J. J. Tregoning, "Composition and Physical Properties of Aqueous Extract From Portland Cement Clinker Pastes Containing Added Materials," *Journal of Research, Nat. Bureau Standards*, Vol. 25, P. 215 (1943). (R. P. 15 30.)

TABLE VII.—RESULTS OF PHYSICAL TESTS FOR CEMENTS OF CLINKER G.

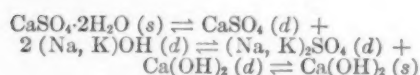
SO <sub>2</sub> Content, per cent	Normal Con- sistency	Setting Time Gillmore		Compressive Strength, psi., at Age Indicated <sup>a</sup>			Length Change of Mortar Prisms at 7 Days, per cent	
		Initial	Final	1 day	3 day	7 day	Expansion in Water <sup>b</sup>	Contraction in Air <sup>b</sup>
SO <sub>2</sub> FROM GYPSUM								
2.50.....	26.0	4:15	6:20	1578	3738	5358	0.008	0.070
2.75.....	26.0	4:10	6:15	1768	3967	5584	0.005	0.067
3.00.....	26.0	4:00	6:10	1728	3683	5333	0.005	0.065
3.25.....	25.5	4:25	6:15	1745	3801	5558	0.007	0.076
3.50.....	25.5	4:00	6:00	1720	3888	5417	0.006	0.064
SO <sub>2</sub> FROM ANHYDRITE								
2.50.....	28.0	1:35	6:00	1069	3153	5008	...	...
2.75.....	28.0	1:35	5:55	1310	3442	5154	...	...
3.00.....	28.0	1:45	6:00	1477	3384	4983	...	...
3.25.....	27.5	1:50	6:05	1639	3613	5225	...	...
3.50.....	27.0	3:15	6:00	1858	3758	5292	...	...
SO <sub>2</sub> FROM MIXTURES OF ANHYDRITE PLUS GYPSUM								
3.25 (a) <sup>c</sup> ...	25.0	3:30	6:15	2135	3533	5817	0.010	0.071
3.25 (b) <sup>c</sup> ...	25.5	3:30	6:25	1979	4233	5733	0.022	0.060
3.25 (c) <sup>c</sup> ...	26.0	3:40	6:35	2001	4167	5650	0.010	0.079
3.25 (d) <sup>c</sup> ...	26.0	3:45	6:35	1930	3992	5542	-0.011	Broken

<sup>a</sup> A.S.T.M. Method C 109-47 mortar.<sup>b</sup> Expansion during 6 days in water after 1 day in mold; contraction during 7 days in laboratory air after 1 day in mold and 6 days in water.<sup>c</sup> (a) = 25.0 per cent anhydrite; (b) = 37.5 per cent anhydrite; (c) = 50.0 per cent anhydrite; and (d) = 62.5 per cent anhydrite.

TABLE VIIA.—SO<sub>3</sub> CONTENT OF EXTRACTS FROM MORTAR SPECIMENS AT AGE INDICATED, G. PER LITER.

SO <sub>3</sub> FROM SOURCE INDICATED										
SO <sub>3</sub> Con- tent, per cent	Gypsum		Anhydrite		Mixtures of Anhydrite and Gypsum Containing 3.25 per cent SO <sub>3</sub> with per cent of Anhydrite Indicated					
					37.5		50.0		62.5	
	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.	18 hr.	24 hr.
2.50..	0	0	0	0	...	...	...	...	...	...
2.75..	0	0	0	0	...	...	...	...	...	...
3.00..	0.07	0	0	0	...	...	...	...	...	...
3.25..	0.32	0	0	0	0	0	0	0	0	0
3.50..	0.65	0.10	0	0	...	...	...	...	...	...

alkali hydroxides and sulfates. It is known that, in cement clinker, a portion of the alkalis is in forms which dissolve very rapidly in the liquid phase of the cement pastes and that a portion is in forms which are released to the liquid phase gradually as the cement minerals react with water. Alkalies present in the clinker as sulfates dissolve as such; those present as glass and combined in compounds with silica and alumina probably are released as hydroxides by hydrolysis of these materials. The calcium silicates and aluminates hydrolyze to form calcium hydroxide, and the calcium sulfate dissolves as such. These products then react<sup>9</sup> with each other to establish the equilibrium represented below in which (s) = solid and (d) = dissolved.



All cements contain many times the amount of calcium sulfate which is soluble in the amount of water used in practice. Hence, the solid calcium sulfate dissolves gradually to maintain the equilibrium represented above as the aluminates tend to destroy it by combining with the SO<sub>3</sub> in the solution to form calcium sulfoaluminate.

It is evident that, as the reaction of the cement with water proceeds, a time will be reached at which the solid calcium sulfate will be depleted, and that, from this time on, the concentration of SO<sub>3</sub> in the liquid will decrease until it reaches the value required by the solubility of calcium sulfoaluminate. As the concentration of SO<sub>3</sub> decreases, the concentrations of the alkali hydroxides increase until, finally, the liquid phase is almost entirely a solution of calcium and alkali hydroxides.

Lerch,<sup>3</sup> Table VII (p. 1274), shows that, with seven cements from clinker No. 16823 containing from 1.0 to 4.0 per cent SO<sub>3</sub>, the amounts of heat liberated were almost identical for the seven cements at 4 hr. whereas at 12 hr. the

<sup>9</sup> W. C. Hansen and E. E. Pressler, "Solubility of Ca(OH)<sub>2</sub> and CaSO<sub>4</sub>·2H<sub>2</sub>O in Dilute Alkali Solutions," *Industrial and Engineering Chemistry*, Vol. 39, p. 1280 (1947).

TABLE VIII.—HEATS OF HYDRATION IN THE CONDUCTION CALORIMETER AT 70 F.

Samples 1, 2, and 3—Clinker C plus 2.25 per cent SO<sub>3</sub>:

1. Gypsum 100 per cent
2. Gypsum 62.5 per cent, anhydrite 37.5 per cent
3. Gypsum 37.5 per cent, anhydrite 62.5 per cent

Samples 4, 5, and 6—Clinker G plus 3.25 per cent SO<sub>3</sub>:

4. Gypsum 100 per cent
5. Gypsum 62.5 per cent, anhydrite 37.5 per cent
6. Gypsum 37.5 per cent, anhydrite 62.5 per cent

IMMEDIATE HEAT OF HYDRATION FOR SAMPLE INDICATED, CAL. PER G. CEMENT

Minutes	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
2.....	2.61	2.78	2.61	1.78	2.78	2.89
5.....	3.05	3.30	3.19	3.10	4.08	4.31
10.....	3.32	3.59	3.52	3.62	4.53	4.68
20.....	3.58	3.79	3.84	3.88	4.92	5.09
30.....	3.71	3.89	3.97	4.08	5.11	5.27

CUMULATIVE HEAT OF HYDRATION, CAL. PER G. CEMENT

Hours	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
1.....	4.1	4.4	4.4	4.7	5.9	6.0
2.....	4.5	4.8	4.9	5.2	6.5	6.5
4.....	5.4	5.9	6.0	6.3	7.9	8.2
6.....	7.1	7.8	8.0	9.0	10.3	11.7
8.....	10.1	11.1	11.3	14.3	15.9	17.7
10.....	13.8	15.1	15.3	21.4	22.8	24.9
12.....	17.6	19.4	19.2	28.0	29.7	31.7
14.....	21.3	23.4	22.2	33.9	35.7	37.3
16.....	25.4	26.6	24.8	39.6	41.0	41.9
18.....	29.0	29.6	27.5	44.1	45.4	46.0
20.....	31.1	32.3	30.2	47.4	48.2	48.9
22.....	32.9	34.7	32.9	49.9	50.5	51.0
24.....	34.1	36.3	35.3	51.8	52.4	52.5
30.....	36.9	39.3	39.6	56.2	57.0	56.5
36.....	39.1	41.4	42.0	59.5	60.1	59.7
42.....	41.3	43.5	44.2	62.0	62.7	62.5
48.....	43.3	45.5	46.2	64.5	65.1	64.9
54.....	45.3	47.4	48.4	67.2	67.4	67.5
60.....	47.2	49.5	50.6	69.7	69.3	70.0
66.....	49.0	51.4	52.5	71.8	71.3	72.2
72.....	50.8	53.4	54.5	73.8	72.8	74.4

amounts were 19.4, 37.4, 44.3, and 51.0 calories per g. for SO<sub>3</sub> contents of 1.0, 1.5, 1.9, and 2.4 per cent, respectively. These data show the marked effect of increased SO<sub>3</sub> upon the rate of reaction of this cement. From what was said immediately above, during the first few hours the liquid phase in the pastes of these cements would tend to have identical compositions since the clinkers

drite is to replace gypsum without decreasing the early strength possible from a cement with the optimum amount of gypsum, it seems that the natural anhydrite would have to have the ability to maintain a high concentration of SO<sub>3</sub> in the liquid phase of the cement paste over the period of time during which a similar concentration of SO<sub>3</sub> is maintained with gypsum. It was of interest,

TABLE IX.—CONCENTRATIONS OF SO<sub>3</sub> IN THE LIQUID PHASE OF CEMENT PASTE AT ROOM TEMPERATURE

	Concentration of SO <sub>3</sub> at Age Indicated, g. per liter.				
	3 min.	8 hr.	12 hr.	18 hr.	24 hr.
Clinker A with 2.0 per cent SO <sub>3</sub> as gypsum.....	1.51	1.55	1.53	0.49	0.21
Clinker A with 1.0 per cent SO <sub>3</sub> as gypsum + 1.0 per cent SO <sub>3</sub> as anhydrite.....	1.60	1.56	0.95	0.64	0.27
Clinker A with 2.0 per cent SO <sub>3</sub> as anhydrite.....	1.65	1.26 <sup>a</sup>	...	...	...
Clinker B with 2.0 per cent SO <sub>3</sub> as gypsum.....	2.62	2.43	2.54	1.90	1.03
Clinker B with 1.0 per cent SO <sub>3</sub> as gypsum + 1.0 per cent SO <sub>3</sub> as anhydrite.....	2.57	2.40	1.60	1.86	1.49
Clinker C with 2.0 per cent SO <sub>3</sub> as gypsum.....	1.45	1.44	0.23	0.00	0.01
Clinker C with 1.0 per cent SO <sub>3</sub> as gypsum + 1.0 per cent SO <sub>3</sub> as anhydrite.....	1.42	0.74	0.17	0.01	0.01

<sup>a</sup> Values at 2 and 4 hr. were 1.64 and 1.49, respectively.



therefore, to determine the extent to which the concentration of  $\text{SO}_3$  in the liquid phase of a cement paste was changed by replacing the gypsum in the cement with natural anhydrite.

In this study, pastes of 100 g. of cement and 75 ml. of distilled water were rotated in stoppered flasks at room temperature for various periods of time. These were then filtered on a Büchner funnel and the filtrates were analyzed for  $\text{SO}_3$ . The results of this study are given in Table IX.

Before discussing these data, the use of pastes of this type should be justified because, in the past, many investigators have felt that results of studies with pastes having water-cement ratios different from those of practice could not be interpreted in terms of concrete. However, since it is now known, as pointed out previously, that the liquid phase of a cement paste is essentially a solution of calcium and alkali hydroxides and sulfates, it should be possible to predict the effect of changing the water-cement ratio. The concentrations in the liquid phase of the paste depend primarily upon the amount of readily soluble alkalis in the clinker, as may be seen from the work of Hansen and Pressler<sup>9</sup> which gives the solubilities of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  and  $\text{Ca}(\text{OH})_2$  in solutions of  $\text{KOH}$  and  $\text{NaOH}$ . Since the amount of alkalis dissolved in the early periods will not vary greatly with the water-cement ratio of the paste, the major effect of using a higher water-cement ratio than that normally used in practice will be to dilute the solution with respect to sodium and potassium salts and to decrease their concentrations in the solutions. The principal effect of this will be to decrease the concentration of calcium sulfate in the solution in the same proportions. If the rate of reaction of the cement varies with the concentrations of the calcium and alkali hydroxides and sulfates in the liquid phase, changes in the water-cement ratio of the paste will change the rate of reaction, but changes in the water-cement ratio within certain limits should not change the nature of the reaction products because of their relatively low solubilities.

The data in Table IX indicate that the

rates of reaction in these pastes were appreciably slower than they were in the mortars used in the physical tests. For example, the data for the extracts from the mortars made with clinker B ground with 2.0 per cent  $\text{SO}_3$  as gypsum indicated that the  $\text{SO}_3$  had been combined almost completely in 24 hr. whereas the results in Table IX show a relatively large amount of  $\text{SO}_3$  uncombined at 24 hr. This substantiates the conclusion given above from the results by Lerch that the rate of reaction is dependent upon the concentration of  $\text{SO}_3$  of the solution.

Again, from the data in Table IX, it may be seen that the concentrations of  $\text{SO}_3$  in the pastes during the early periods varied between clinkers. This is expected since, as pointed out previously, the solubility of gypsum varies with the alkali content of the solution which, in turn, varies with readily soluble alkali content of the clinker. In some cases, the concentrations of  $\text{SO}_3$  were higher at 3 min. than they were at 8 hr. With clinker A, the concentrations of  $\text{SO}_3$  were higher at 3 min. in the pastes of cements with a mixture of gypsum and anhydrite and anhydrite alone than they were in the cement with gypsum alone. These variations probably are the result of supersaturation because the fine broken fragments of gypsum and anhydrite would tend to produce supersaturated solutions during the first few minutes.

With clinkers A and B, the mixtures of equal parts of anhydrite and gypsum were able to maintain the concentrations of  $\text{SO}_3$  equal to those obtained with gypsum alone for periods of at least 8 hr. This is the period during which the cements obtained their initial and final sets, and, as may be seen from Table I and II, replacing gypsum by anhydrite up to 62.5 per cent anhydrite had no significant effect upon the setting times of these cements.

The optimum amount of  $\text{SO}_3$  selected for clinker C was 2.75 per cent and, from Table IX, it may be seen that this clinker combined with  $\text{SO}_3$  at a much faster rate than did clinkers A and B. Also, from Table IX, it appears that, although the mixture of anhydrite and

gypsum was not able to maintain the concentration of  $\text{SO}_3$  in the liquid phase obtained with gypsum, the mixture of anhydrite and gypsum was combined with the aluminates at about the same time as was the gypsum alone.

The results of these studies of the extracts of cement pastes show that mixtures of equal parts of anhydrite and gypsum behave very much like gypsum alone with respect to rate of solution and rate of reaction with cement minerals. Accordingly, they explain why mixtures of anhydrite and gypsum are as effective in controlling the rates of setting and hydration of certain cements as is gypsum alone.

#### SUMMARY

The results of this study show that, with a number of portland cements, mixtures of anhydrite and gypsum could replace gypsum without adversely affecting either the setting times of the cement pastes or the strength and volume change characteristics of the concrete made from them. With six of the seven clinkers studied in this investigation, amounts of anhydrite equivalent to from 62.5 to 71.5 per cent of the  $\text{SO}_3$  in the cement yielded satisfactory results. Anhydrite, equivalent to more than 25 per cent of the  $\text{SO}_3$  content of the cement, appeared to be harmful with the one clinker.

Since this study was limited to seven clinkers, general conclusions for all clinkers cannot be made. However, it appears likely that, with very few exceptions, 25 to 75 per cent of the gypsum could be replaced with natural anhydrite. In a few cases, it might be possible to substitute anhydrite completely for gypsum.

#### Acknowledgment:

The authors take pleasure in acknowledging the assistance of their associates in the research laboratories of their company who contributed to this work. They are especially grateful to Mr. O. A. Ellingson, Mr. L. L. Huspek, and Mr. H. O. Nickelsen, who supervised the chemical work, physical testing and heat of hydration studies, respectively.

#### DISCUSSION

range of compositions investigated. The basic clinkers used are all in the low-alkali range and, also, most of them have relatively low potential tricalcium aluminate contents. Such clinkers are usually much more tolerant of the rate of solution of the retarder than those with higher alkali and tricalcium aluminate content.

Difficulty with set arises when the rate of solution of tricalcium aluminate in cement exceeds the rate of solution of the calcium sulfate. The lower solubility of anhydrite in comparison with gypsum is well known. The action of alkali in increasing the rate of hydration of tricalcium aluminate has also been recognized by L. Forsen and others. With

Mr. M. A. SWAYZE.<sup>1</sup>—Messrs. Hansen and Hunt have presented an excellent paper on use of anhydrite in portland cement. However, a note of warning should be sounded for the benefit of those who might apply the conclusions to production of cement outside the

<sup>1</sup> Director of Research, Lone Star Cement Co., New York, N. Y.

Type I cements, where the tricalcium aluminate content is at or above 13 per cent or so and the alkali content exceeds 0.7 to 0.8 per cent, I would predict great danger of quick setting in the cement if anhydrite is substituted for gypsum as retarder.

Another point in favor of gypsum as a retarder in comparison with anhydrite is its action as a grinding aid. The authors inform me that the data are based on laboratory grinds. Our own records of similar tests show that gypsum increases the grindability of clinker in laboratory mills, whereas use of either anhydrite or plaster of Paris has no such effect. If a record were made of the number of mill revolutions required to attain the desired fineness, the data would be of interest to cement manufacturers concerned with grindability of various types of cement.

The purpose of these remarks is to warn that substitution of anhydrite for all or even part of the gypsum should not be attempted unless an adequate study of the effect of the substitution on stability of setting time and on grindability has been made, as the authors have done.

MR. PAUL S. ROLLER<sup>2</sup> (by letter).—I believe that the authors' conclusions in regard to the use of anhydrite in portland cement should be taken guardedly. They have overlooked an important variable in this problem, and this is all the more surprising since this variable was fully considered in a paper on this subject, which the authors have not referred to, entitled "Relative Value of Gypsum and Anhydrite as Additions to Portland Cement" by the writer and Murray Halwer, published as *Technical Paper 578*, Bureau of Mines, 1937.

The important variable in this system is the adsorbed water vapor in the cement. In this paper, and in previous publications of the writer, it is shown that the retardation of the setting of the clinker is profoundly affected by the adsorption. Only a few tenths of a per cent of adsorption of water vapor may determine whether the clinker will be slow or quick setting, and whether or not anhydrite can be used, and the percentage which may be incorporated as a replacement of gypsum.

On the basis of the observations which have been made, I conclude that the action of a retarder of portland cement consists of two parts. One of these involves the chemical effect of the retarder, residing chiefly in the calcium ion reaction, and the other the contribution of adsorbed water vapor which it can make to the clinker. In regard to the latter point, one of the functions of added gypsum is to provide water vapor to the clinker at a time of great recep-

tivity during the ball mill grinding. To the extent that this reaction is important, one cannot envisage the complete replacement of gypsum by anhydrite as the possibility implied by Hansen and Hunt. In *Technical Paper 578* we concluded that a 25 per cent replacement of gypsum by anhydrite would be generally possible, and frequently it could be up to 50 per cent. In my opinion gypsum is favored not only because of its water-vapor contributing capacity, but also its greater rate of solution and its greater solubility when in the partially dehydrated state in which it for the most part probably exists in cement. The added gypsum provides the necessary reactions in trigger-like fashion, permitting the slower-acting anhydrite to exert its effects on the cement later on.

MR. W. C. HANSEN (author's closure).—Mr. Swayze points out that it might not be possible to substitute anhydrite for gypsum with certain cements. We would not expect from the results of our study to find very many cements which would not perform satisfactorily with gypsum containing a considerable percentage of anhydrite. However, we agree with Mr. Swayze that each plant should determine that the substitution of anhydrite for a part or all of the gypsum has no harmful effect upon the properties of the cement before deciding to make such a change.

Mr. Swayze raises a question regarding the influence of the type of calcium sulfate on the grindability of the clinker. In our study, each clinker was ground with gypsum to give cements with 0.5 and 3.5 per cent  $\text{SO}_3$  and with anhydrite to give cements with 0.5 and 3.5 per cent  $\text{SO}_3$ . Records were kept of the number of mill revolutions for each grind. It is not possible from these data to find a consistent difference between the number of revolutions required to give the desired specific surface and the quantity of calcium sulfate in the cement or the type of calcium sulfate used. For example, with one clinker, specific surfaces of  $1825 \pm 25$  sq. cm. per g. were obtained at 2400 revolutions with 3.5 per cent  $\text{SO}_3$  as gypsum, at 2350 revolutions with 3.5 per cent  $\text{SO}_3$  as anhydrite, at 2250 revolutions with 0.5 per cent  $\text{SO}_3$  as gypsum, and at 2200 revolutions with 0.5 per cent  $\text{SO}_3$  as anhydrite. In this series of tests, one might conclude that the grinding efficiency decreased with increasing amounts of calcium sulfate and with substitution of anhydrite for gypsum. However, it is believed that such a conclusion would not be justified from the results of a single series of tests in a small batch mill, but one might be justified in the conclusion that the amount and type of calcium sulfate had no effect upon the efficiency of grinding.

The authors are familiar with some work in which a number of different clinkers were ground, both with and without gypsum in porcelain jar mills. In all cases, it was difficult to get the desired surface in the grinds without gypsum because of the tendency of the cement to coat the balls and sides of the mill, whereas no such coating was observed when gypsum was used. However, in our studies, in which the grinding was done in a steel mill, no coating was observed in any of the grinds.

It is impossible from our studies to predict that substitution of anhydrite for gypsum will have an effect upon the grindability of the cement. However, this work was limited to seven clinkers and to grinding in a small batch mill. The fact that Mr. Swayze has found differences in grindability when either anhydrite or plaster of Paris was substituted for gypsum in laboratory tests points to the possibility that such differences might be found under some conditions in commercial practice. Accordingly, any plant contemplating the substitution of anhydrite for gypsum should, as Mr. Swayze points out, recognize this possibility and determine whether or not such a substitution would have an adverse effect upon the efficiency of grinding.

Mr. Roller calls attention to the part played by water vapor in the setting of cements. We were familiar with the very valuable publications of Roller and his coworkers on this and other phases of the setting of cement. A review of the literature was not given because of the changes which have been made in the composition and the manufacture of cement over the years and because Lerch's work has shed new light on the role played by calcium sulfate in the setting and hardening of portland cement.

Mr. Roller emphasizes the ability of gypsum to liberate water vapor to the clinker during grinding and of producing calcium sulfate hemihydrate and soluble anhydrite which dissolve at high rates. In the manufacture of cement today, efforts are made to prevent as far as possible dehydration of the gypsum because it is recognized that dehydrated gypsum produces what is termed false set or premature stiffening.<sup>3</sup> The means of minimizing dehydration of the gypsum are cooling the clinker prior to grinding, cooling the mills during grinding, adding small amounts of water to the mills, and cooling the cement prior to storage. We believe, therefore, that the ability of gypsum to provide water vapor during grinding is no longer a reason for using gypsum in cement.

<sup>3</sup> "Lea and Dresch, the Chemistry of Cement and Concrete," Edward Arnold and Co., p. 188.

<sup>2</sup> Chemist, Hyattsville, Md.



## Paint and Varnish Technology

To THE industrial user and the paint manufacturer this comprehensive volume edited by William von Fischer, Head, Dept. of Chemistry and Chemical Engineering, Case Institute of Technology, should be very useful. It presents a survey of the chemistry, formulation, manufacture, and applications of paints, varnishes, lacquers, and enamels. Sufficient chemistry is included to indicate the derivation of synthetic resin bases and the mechanism of the action of drying oils, driers, solvents, and other raw materials. Throughout the book the emphasis is on technological aspects, and information on exposure tests, viscosity, film properties, hiding power, special applications and surface preparations is given. There are illustrations, charts, tables of data, and bibliographic references.

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## Standard Welding Terms and Chart of Welding Processes

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NEARLY 25 years ago the American Society for Testing Materials and the Institute of Petroleum agreed to standardize on the spelling of the word "KEROSENE" with the suffix "INE." It was agreed that in petroleum nomenclature the ending "ENE" is generally applied to pure hydrocarbons. Furthermore, since the suffix "INE" is applied to gasoline it should also be applied to kerosene.

The American Society for Testing Materials and the Institute of Petroleum, realizing that there is still not uniformity in the petroleum world regarding the correct spelling of this particular product, desire to reiterate their strong recommendation for international recognition of the spelling KEROSENE.

## Indentation Hardness Testing

As A result of over 20 years of experience in hardness testing—and because of a great number of inquiries which have come to his attention through A.S.T.M. committee work, conferences, lectures, correspondence and other work with the American Society for Metals and American Society for Tool Engineers—Mr. Vincent E. Lysaght has written this book.

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Vincent E. Lysaght is a graduate of M.I.T. Since 1926 he has been associated with the Wilson Mechanical Instrument Company, where he has dealt with all problems involving testing, development, and research in hardness testing. He has written many articles and presented talks on the subject to many chapters of the A.S.M., A.S.T.M., and A.S.T.E.

For \$5.50, "Indentation Hardness Testing" can be obtained from the Reinhold Publishing Corp., Dept. 108, 330 West 42nd St., New York 18, N. Y.

## Economic Effects of Standardization Pervade Our Industrialized Society

THE following is the summary included as the last portion of a paper published in the Spring, 1949, *Standards World* by F. E. Powell entitled "Economic Aspects of Standardization." Mr. Powell is a Technologist, Commodity Standards Division, National Bureau of Standards. In his paper he makes pertinent reference to A.S.T.M.

### SUMMARY

"To summarize, we find that the economic effects of standardization pervade our industrialized society. They are evident not only in physical production for the mass market but in the intangible forms of industrial process and our common speech. The essence of our study, with particular reference to mass production and merchandizing, may be outlined as follows:

"1. Interchangeability, broadly interpreted, is the basic principle of standardization and provides a criterion for gaging both its success and its economic worth. The principle of interchangeability applies as well to close functional as to close physical similarity, as illustrated by transactions based on performance specifications. The degree of interchangeability varies widely. Stated generally as the ratio of actual to potential use of a

standard item, it affords a measure for standardization progress as well as a reference basis for setting the optimum degree of application.

"2. Standardization provides strong support to our system of free enterprise which was classically founded on moderate-scale competition in goods having supposedly identical units. Adherence to specific standards now makes competition possible on the large scale found on the stock and commodity exchanges. Today's standard products are actually interchangeable to a greater degree than the 'standard' goods of the classical free enterprise economists. On any scale of trading, standards provide buyer and seller alike with definite bases of comparison and support reputable producers by setting specific goals to attain or exceed.

"3. Historically, standardization and business in this country grew up through mutual voluntary support. Now a large industry in its own right, standardization has not only made mass production possible but has provided direct savings amounting to millions of dollars on individual programs. Similar benefits are expected from international standardization now under way, notably the unification of American, British, and Canadian screw threads. The field of national as well as international standardization offers us broad and challenging opportunity for improving our economic well-being."

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